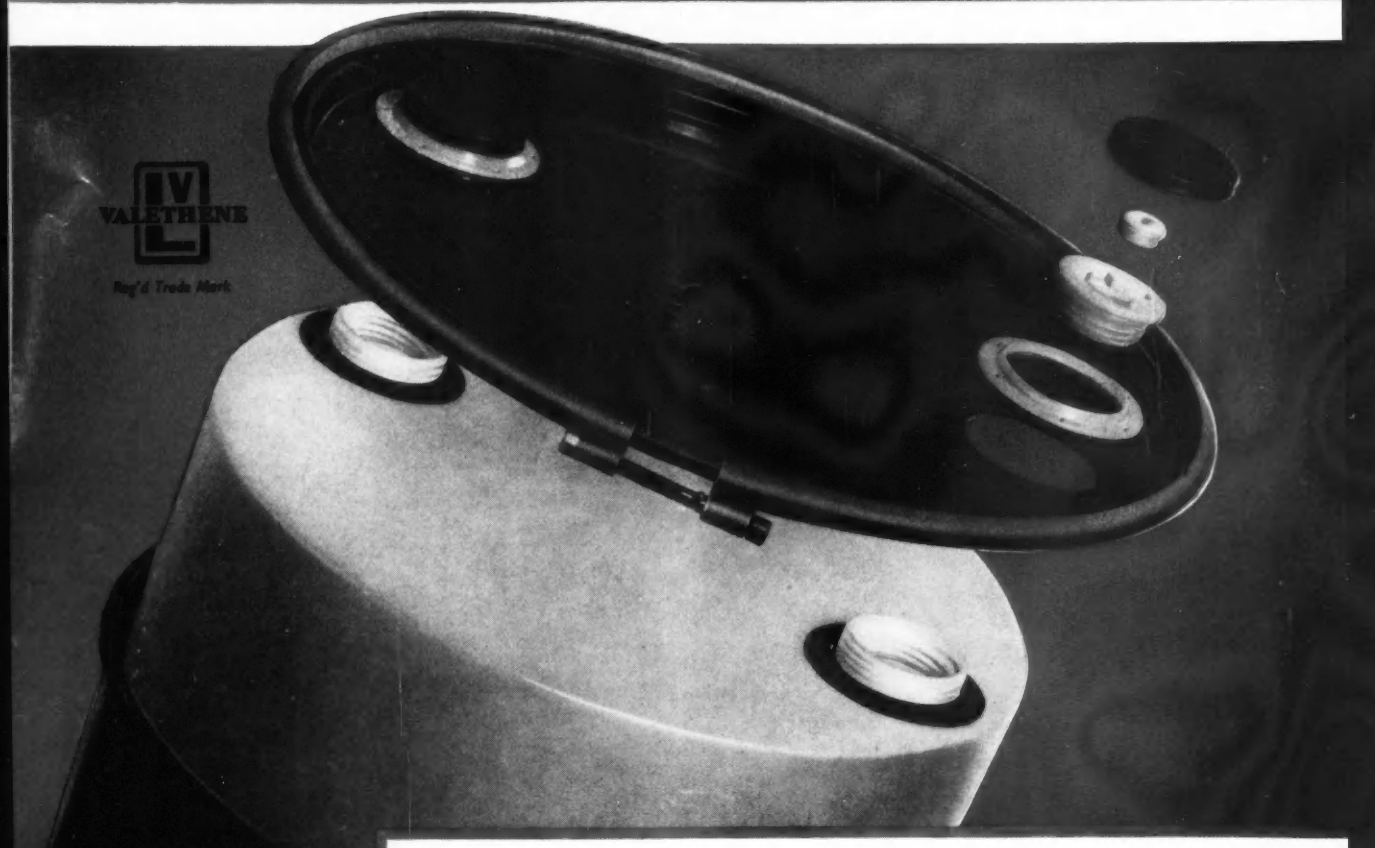
Chemical Age

GOVERNMENT AID FOR WATER RESEARCH
(page 853)

VOL. 81 No. 2080

23 May 1959



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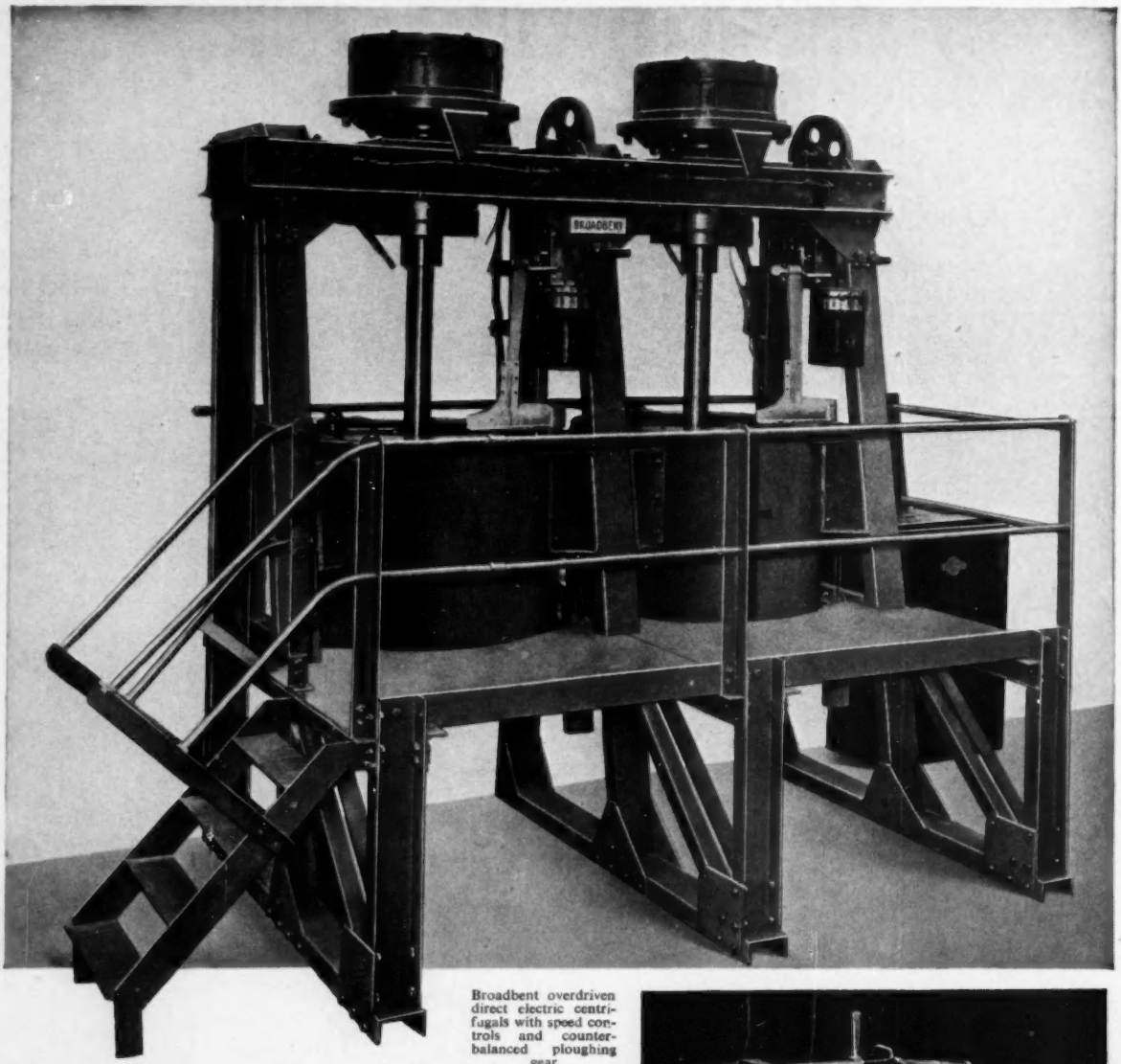

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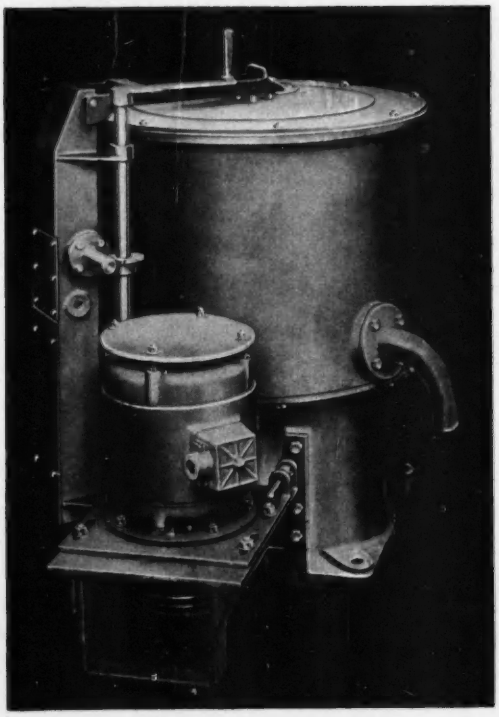

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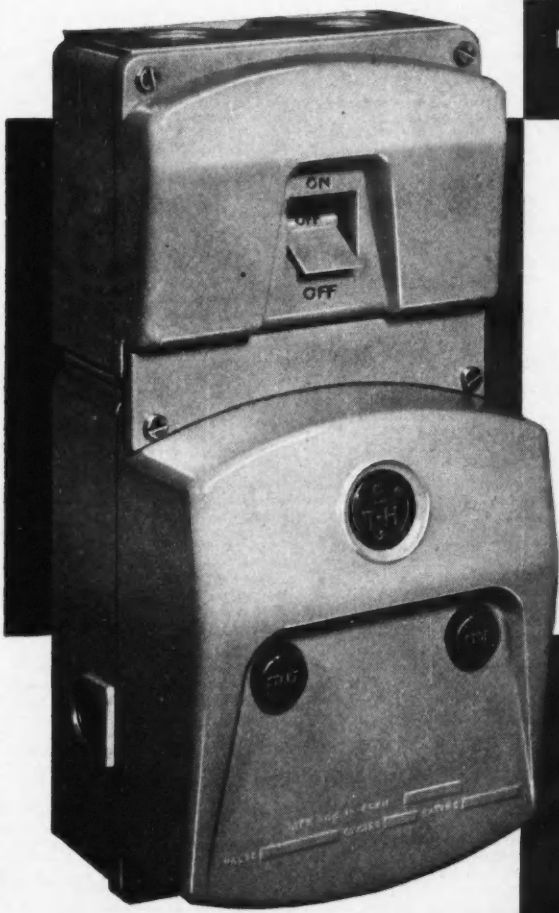

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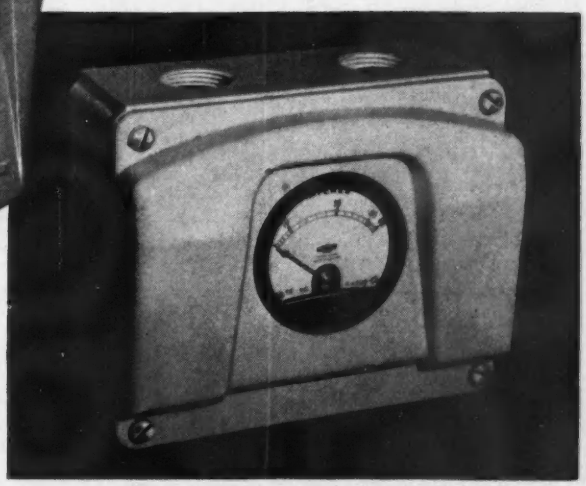

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INDEX TO ADVERTISERS

The first figures refer to advertisements in **Chemical Age Directory & Who's Who,** *the second to the current issue*

Page		Page
160	A.P.V. Co. Ltd., The	—
154	Acalor (1948) Ltd.	—
85	Accrington Brick & Tile Co. Ltd., The	—
	African Pyrethrum Technical Information Centre Ltd.	—
117	Air Trainers Link Ltd.	—
131	Albany Engineering Co. Ltd., The	—
	Albright & Wilson (Mfg.) Ltd.	—
	Alchemy Ltd.	848
86	Alginate Industries Ltd.	—
100	Allen, Edgar, & Co. Ltd.	—
118	Allen, Frederick (Poplar) Ltd.	—
	Alumina Co. Ltd., The	—
	Amalgamated Oxides (1939) Ltd.	—
	American Cyanamid	—
102	Anglo-Dal Ltd.	—
	Anthony, Mark & Sons Ltd.	872
166	Armour Chemical Industries Ltd.	—
G/Card	Ashmore, Benson, Pease & Co. Ltd.	—
	Ashworth, Arthur, Ltd.	—
	Associated Electrical Instruments Ltd. Turbine-Generator Division	—
103	Associated Lead Mfrs. Ltd.	—
G/Card	Audley Engineering Co. Ltd.	—
169	Automotive Products Ltd.	—
	Avo Ltd.	—
	Baird & Tatlock Ltd.	—
143	Baker Perkins Ltd.	—
	Baldwin Instrument Co.	—
159	Balfour, Henry, & Co. Ltd.	—
	Balfour Group of Companies, The	—
120	Barclay Kellett & Co. Ltd.	—
	Bennett, H. G., & Co. (Gloves) Ltd.	—
87	Bennett, Sons & Shears Ltd.	—
G/Card	Berk, F. W., & Co. Ltd.	—
104	Bivac Air Company Ltd.	—
132	Black, B., & Son Ltd.	—
2	Blackman, Keith, Ltd.	—
197	Blundell & Crompton Ltd.	—
	Borax & Chemicals Ltd.	—
84	Borax Consolidated Ltd.	—
	Borer Engineering Co. Ltd.	—
4	Boulton, William, Ltd.	—
97	Bourne Chemical Industries Ltd.	—
	Bowmans Chemicals Ltd.	—
119 & 147	Braby, Frederick, & Co. Ltd.	—
86	Bristol Piping Co. Ltd., The	—
	British Acheson Electrodes Ltd.	—
	British Carbo-Norit Union Ltd.	—
	British Ceca Co. Ltd., The	871
	British Celanese Ltd.	845
	British Drug Houses Ltd., The	—
	British Dyewood Co. Ltd., The	—
146	British Ermeto Corporation Ltd.	—
Spine	British Geon Limited	—
	British Industrial Solvents	—
220	British LaBour Pump Co. Ltd.	—
	British Lead Mills Ltd.	—
Spine	British Resin Products Ltd.	—
132	British Rototherm Co. Ltd., The	—
89	British Steam Specialties Ltd., The	—
134	British Tar Products Ltd.	—
	British Thomson-Houston Co. Ltd., The	843
231	British Titan Products Co. Ltd.	—
267	Broadbent, Thomas, & Sons Ltd.	Cover ii
137	Brotherhood, Peter, Ltd.	—
	Brough, E. A., & Co. Ltd.	—
101	Bryan Donkin Co. Ltd., The	—
	Burndept Ltd.	—
132	Burnett & Rolfe Ltd.	—

Page		Page
162	Bush, W. J., & Co. Ltd.	—
	Buss Ltd.	—
88	Butterfield, W. P., Ltd.	—
	Butterworths Scientific Publications	—
126	Calder Vale Glassworks Ltd.	—
	Callow Rock Lime Co. Ltd., The	—
	Cambridge University Press	—
	Cannon (CP) Ltd.	—
	Carbon Dioxide Co., The	—
	Catterson-Smith, R. M., Ltd.	—
210	Causeway Reinforcement Ltd.	—
	Chapman & Hall Ltd.	—
	Chemical Age Enquiries	877 & 878
178	Chemical Construction (G.B.) Ltd.	—
	Chemical & Insulating Co. Ltd., The	851
106	Chemical Workers' Union	—
	Chemicals & Feeds Ltd.	—
	Chemidus Plastics Ltd.	—
	Chemitrade Ltd.	—
	Christy & Norris Ltd.	—
	Ciba (A.R.L.) Ltd.	—
110	Ciba Clayton Ltd.	—
	Ciech Ltd.	Cover iii
	City Engineering Co. (Boreham Wood) Ltd.	—
	Clark Ltd.	—
	Classified Advertisements	875 & 876
95	Clayton, Son & Co. Ltd.	—
142	Clydesdale Chemical Co. Ltd., The	848
	Cochran & Co. (Annan) Ltd.	—
	Cohen, George, Sons & Co. Ltd.	—
121	Cole, R. H., & Co. Ltd.	—
90	Collins Improved Firebars Ltd.	—
	Colt Ventilation Ltd.	—
	Colvin-Smith Ltd.	—
133	Comet Pump & Eng. Co. Ltd., The	—
	Constable & Co.	—
	Constructors, John Brown, Ltd.	—
	Contoura Photocopying Ltd.	—
	Controlled Convection Drying Co.	—
	Crofts (Engineers) Ltd.	—
	Cromil & Piercy Ltd.	—
	Crosfield, Joseph, & Sons Ltd.	—
99	Cruickshank, R., Ltd.	—
214	Curran, Edward, Engineering Ltd.	—
171	Cyanamid of Great Britain Ltd.	—
88	Cyclops Engineering Co. Ltd., The	—
	Cygnet Joinery Ltd.	—
	Dalglish, John, & Sons Ltd.	—
150	Danks of Netherton Ltd.	—
149	Davey & Moore Ltd.	—
173	Davey, Paxman & Co. Ltd.	—
	Dawson, McDonald & Dawson Ltd.	—
	Degenhardt & Co. Ltd.	—
94	Derby Luminescents Ltd.	—
175	Distillers Co. Ltd., The (Chemical Div.)	—
	Distillers Co. Ltd., The (Engineering Div.)	—
163	Dorr-Oliver Co. Ltd.	—
139	Doulton Industrial Porcelains Ltd.	—
	Dowlow Lime & Stone Co. Ltd., The	—
136	Dring & Fage Ltd.	—
183	Drummond Patents Ltd.	—
151	Dryden, T., Ltd.	—
	Dupree Swift & Co. Ltd.	—
96	E.C.D. Ltd	—
	Electro-Chemical Engineering Co. Ltd.	—
	Electro Chemical Engineering Co. Ltd.	—
	Electronic Switchgear (London) Ltd.	—
	Electrothermal Engineering Ltd.	—
	Elga Products Ltd.	—

Page		Page
	Elliott, H. J., Ltd.	—
158	Elmatic	—
	Enamelled Metal Products Corporation (1933) Ltd.	—
	Engelhard Industries Ltd. (Hanovia Lamps Division)	—
111	Engelhard Industries Ltd. (Baker Platinum Division)	—
	English Glass Co. Ltd., The	—
G/Card	Erinoid Ltd.	—
	Evans, Joseph, & Sons (Wolverhampton) Ltd.	—
	Evered & Co. Ltd.	—
	Eversheds & Vignoles Ltd.	—
	Extrudex Ltd.	—
90	Farnell Carbons Ltd.	—
	Fawcett, Preston & Co. Ltd.	—
124	Feltham, Walter H., & Son Ltd.	—
	Ferraris, Fred. Ltd. (Clerkenwell)	—
138	Ferris, J. & E., Ltd.	—
130	Film Cooling Towers (1925) Ltd.	—
	Fisher Governor Ltd.	—
	Foamite Ltd.	—
	Ford, T. B., Ltd.	—
	Foster Instrument Co. Ltd.	—
93	Foxboro-Yoxall Ltd.	—
	Foyle, W. & G. Ltd.	—
	Freeman Taylor Machines Ltd.	—
198	Fuller's Earth Union Ltd., The	—
	Gallenkamp, A., & Co. Ltd.	—
	Gas Council, The	—
	General Electric Co. Ltd.	—
134	Glebe Mines Ltd.	—
218	Goodyear Pumps Ltd.	—
	Gowlands Ltd.	—
107	Graviner Mfg. Co. Ltd.	—
109	Grazebrook, M. & W., Ltd.	—
122	Greeff, R. W., & Co. Ltd.	—
	Grindley & Co. Ltd.	—
138	Haller & Phillips Ltd.	—
124	Harris (Lostock Gralam) Ltd.	874
	Hartley Electromotives Ltd.	—
	Hathernware Ltd.	—
	Haworth, F. (A.R.C.), Ltd.	—
144	Heathway Machinery Co. Ltd.	—
	Herbert, Alfred, Ltd.	—
	High Pressure Components Ltd.	—
	Hilger & Watts Ltd.	—
183	Holland, B. A., Eng. Co. Ltd., The	—
	Hopkin & Williams Ltd.	—
	Humphreys & Glasgow Ltd.	—
6	Huntington, Heberlein & Co. Ltd.	—
	I.C.I. Billingham	—
	I.C.I. Catalysts	—
	I.C.I. General Chemicals Solvents	—
	I.C.I. Ltd. Heavy Organic Chemicals	—
	I.C.I. Metals Titanium D.	—
	I.C.I. Plastics—Darvic	—
	I.C.I. Plastics—Fluon	—
	I.C.I. Ltd. (Plastics Division), Corvic	—
	I.C.I. (Florube) Ltd.	—
	Industrial Descaling Tools Ltd.	—
	Industrial Tapes Ltd.	—
	Infra Red Development Co. Ltd., The	—
	International Combustion Group	—
127	International Furnace Equipment Co. Ltd., The	—

(continued on page 846)

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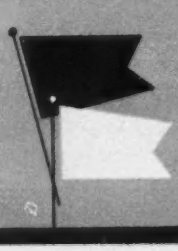

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INDEX TO ADVERTISERS

The first figures refer to advertisements in **Chemical Age Directory & Who's Who,** *the second to the current issue*

Page		Page
	Interscience Publishers Ltd.	—
	Isopad Ltd.	—
102	Jackson, J. G. & Crockatt Ltd.	—
	Jacobson Van Den Berg & Co. (U.K.) Ltd.	—
125	Jenkins, Robert, & Co. Ltd.	—
	Jenkinson, W. G., Ltd.	—
3	Jobling, James A., & Co. Ltd.	—
	Johnson, Matthey & Co. Ltd.	—
98	Johnsons of Hendon Ltd.	—
108	K.D.G. Instruments Ltd.	—
148	K.W. Chemicals Ltd.	—
	Kaylene (Chemicals) Ltd.	—
	Kellie, Robert & Sons Ltd.	—
	Kellogg International Corporation	—
110	Kernick & Son Ltd.	—
265	Kestner Evaporator & Engineering Co. Ltd.	851
	Kestner Evaporator & Engineering Co. Ltd. (*Keebush*)	—
	Kestner (Industrial Safety) Ltd.	—
130	Kier, J. L., & Co. Ltd.	—
	King, G. W., Ltd.	—
208	Kingsley & Keith Ltd.	—
184	Kleen-e-ze Brush Co. Ltd., The	—
122	Laboratory Apparatus & Glass Blowing Co.	—
224	Lambeth & Co. (Liverpool) Ltd.	—
	Langley Alloys Ltd.	—
112	Lankro Chemicals Ltd.	—
205	Laporte Chemicals Ltd.	—
114	Lavino (London) Ltd.	—
173	Leda Chemicals Ltd.	—
96	Leek Chemicals Ltd.	—
	Lees, Henry, & Co. Ltd.	—
112	Leigh & Sons Metal Works Ltd.	—
	Lennig, Charles, & Co. (Great Britain) Ltd.	852
	Lennox Foundry Co. Ltd.	—
129	Light, L., & Co. Ltd.	—
135	Lind, Peter, & Co. Ltd.	—
118	Liquid Solid Separations Ltd.	—
	Lloyd & Ross Ltd.	—
B/cover	London Aluminium Co. Ltd., The	—
142	London Sand Blast Decorative Glass Works Ltd., The	—
	Longman Green & Co. Ltd.	—
	Longworth Scientific Instruments Co.	—
92	Lord, John L., & Son	Cover iv
	Loughborough Glass Co. Ltd.	—
	McCarthy, T. W., & Sons Ltd.	844
	MacLellan, George, & Co. Ltd.	847
	Maine, B. Newton Ltd.	—
126	Manesty Machines Ltd.	—
177	Marchon Products Ltd.	—
	Marston Excelsior Ltd.	—
108	Matthews & Yates Ltd.	—
	May & Baker Ltd.	—
	Measuring & Scientific Equipment Ltd.	—
	Medway Paper Sacks Ltd.	—
	Mervyn Instruments & Co. Ltd.	—
Front cover	Metal Containers Ltd.	Front cover
	Metafiltration Co. Ltd.	—
G/card	Metalock (Britain) Ltd.	—
126	Metcalf & Co.	—
	Metropolitan-Vickers Electrical Co. Ltd.	Cover iv
148	Middleton & Co. Ltd.	—
	Mills Packard Construction Co. Ltd.	—
	Mine Safety Appliances Co. Ltd.	—
	Mirrlees Watson Co. Ltd., The	—
140	Mirvale Chemical Co. Ltd., The	—
	Mitchell, L. A., Ltd.	—
141	Mitchell Cotts Co. Ltd.	—
	Mond Nickel Co. Ltd., The	849

Page		Page
120	Monkton Motors Ltd.	—
	Monsanto Chemicals Ltd.	—
	Moritz Chemical Engineering Co. Ltd.	—
	Mulberry Co., The	—
	National Industrial Fuel Efficiency Service	—
82	Neckar Water Softener Co. Ltd.	850
115	Negretti & Zambra Ltd.	—
	New Metals & Chemicals Ltd.	—
	Newnes, George, Ltd.	—
	Nicolson, W. B. (Scientific Instruments) Ltd.	—
	Nitrate Corporation of Chile Ltd.	—
	Nordac Ltd.	—
146	Odoni, Alfred A. & Co. Ltd.	—
G/card	Oil & Colour Chemists' Association Operation Britain	—
136	Optical-Mechanical (Instruments) Ltd.	—
	Orr Products Ltd.	—
	Palfrey, William, Ltd.	—
	Pascall Engineering Co. Ltd., The	—
8	Paterson Engineering Co. Ltd., The	—
161	Peabody Ltd.	—
	Penrhyn Quarries Ltd.	—
	Permali Ltd.	—
194 & 232	Permutit Co. Ltd., The	—
G/card	Petrocarbon Developments Ltd., The Petrochemicals Ltd.	—
150	Plastic Filters Ltd.	—
	Platon, G. A., Ltd.	—
154	Podmores (Engineers) Ltd.	—
206	Polypenco Ltd.	—
223	Pool, J. & F., Ltd.	—
	Pott, Cassels & Williamson Ltd.	Cover iii
	Potter, F. W., & Soar Ltd.	—
180	Powell Duffryn Carbon Products Ltd.	—
G/card	Power-Gas Corporation Ltd., The	—
197	Prat-Daniel (Stanmore) Ltd.	—
128	Price Stutfield & Co. Ltd.	—
	Price's (Bromborough) Ltd.	—
	Prodorite Ltd.	—
	Purkis, Williams Ltd.	—
190	Pye, W. G., & Co. Ltd.	—
162	Pyrometric Equipment Co. Ltd., The	—
	Q.V.F. Ltd.	—
	Quickfit & Quartz Ltd.	—
186	Reads Ltd.	—
140	Rediweld Ltd.	—
	Research Utilities Ltd.	—
	Reynolds & Branson Ltd.	—
	Rheem Lysaght Ltd.	—
	Richmond Welding Co. Ltd.	—
	Robinson, F., & Co. Ltd.	—
G/card	Rose, Downs & Thompson Ltd.	—
153 & 188	Dr. Rosin Industrial Research Co. Ltd.	—
124	Rotometer Manufacturing Co. Ltd.	—
	S.I.C. Plastics Ltd.	—
118	S.P.E. Company Ltd.	—
	Saint-Gobain	—
113	Sandiacre Screw Co. Ltd., The	—
	Scientific Design Co. Inc.	—
	Scientific Glass-Blowing Co. Ltd.	—
	Scientific Instrument Manufacturers' Association of Great Britain Ltd.	—
	Scott, Ernest, & Co. Ltd.	—
	Scott, George, & Son (London) Ltd.	—
	Sharples Process Engineers Ltd.	—
	Shaw Petrie Ltd.	—
193	Shell Chemical Co. Ltd.	—
	Shell-Mex & B.P. Ltd.	—

Page		Page
	Shipping Studies Ltd.	844
	Short & Mason Ltd.	—
91	Siebe, Gorman & Co. Ltd.	—
	Siemens Edison Swan Ltd.	—
	Sifam Electrical Instrument Co. Ltd.	—
	Simon, Richard, & Sons Ltd.	—
	Sipon Products Ltd.	—
128	Southern Instruments Computer Division	—
128	Spencer Chapman & Messel Ltd.	—
	Stabilag Co. Ltd., The	—
266	Stanton Instruments Ltd.	—
	Stationery Office, Her Majesty's	—
	Staveley Iron & Chemical Co. Ltd.	—
92	Steel, J. M., & Co. Ltd.	—
	Steel & Cowlishaw Ltd.	—
	Stockdale Engineering Co. Ltd.	—
	Stonehouse Paper & Bag Mills	—
	Streamline Filters Ltd.	—
	Sturge, John & E., Ltd.	—
	Sutcliffe Speakman & Co. Ltd.	—
156	Synthite Ltd.	—
149	"T.P." Chemical Engineering Co Ltd.	—
155	Taylor Rustless Fittings Co. Ltd., The	—
142	Taylor Stainless Metals Ltd.	—
152	Thermal Syndicate Ltd., The	—
	Thermo Plastics Ltd.	—
	Thompson, John (Dudley) Ltd.	—
120	Titanium Metal & Alloys Ltd.	—
	Todd Bros. (St. Helens & Widnes) Ltd.	—
144	Towers, J. W., & Co. Ltd.	873
	Triangle Valve Co. Ltd.	—
210 & 224	Tylors of London Ltd.	—
	Unicone Co. Ltd., The	—
	Unifloc Ltd.	—
	Unilever Ltd.	—
	Union Carbide Ltd.	—
	United Coke & Chemicals Co. Ltd.	—
104	United Filters & Engineering Ltd.	—
G/Card	Universal-Matthey Products Ltd.	—
	Vacu-Blast Ltd.	—
	Van Nostrand Co. Ltd.	—
	Vaughan Crane Co. Ltd.	—
	Voss Instruments Ltd.	—
183	W.E.X. Traders Ltd.	—
	Walker, James, & Co. Ltd.	—
	Walker, P. M., & Co. (Halifax) Ltd.	—
	Wallach Bros. Ltd.	—
105	Waller, George, & Son Ltd.	—
98	Wallis, Charles, & Sons (Sacks) Ltd.	—
123	Ward, Thos. W., Ltd.	—
	Warren-Morrison Ltd.	—
152	Watson, Laidlaw & Co. Ltd.	—
	Wellington Tube Works Ltd.	—
116	Wells, A. C., & Co. Ltd.	—
220	Wengers Ltd.	—
	Wheatoe Ltd.	—
	Whiffen & Sons Ltd.	—
184	Whitaker, B., & Sons Ltd.	—
	White, Child & Beney Ltd.	—
123	Widnes Foundry & Engineering Co. Ltd.	—
202	Wilcox, W. H., & Co. Ltd.	—
136	Wilkinson, James, & Son Ltd.	—
94	Williams & James (Engineers) Ltd.	—
122	Wilson, Edward, & Son Ltd.	—
114	Wood, Harold, & Sons Ltd.	—
156	Worcester Royal Porcelain Co. Ltd., The	—
	Worthington-Simpson Ltd.	—
	Wynn (Valves) Ltd.	—
116	Yorkshire Tar Distillers Ltd.	—
106	Zeal, G. H., Ltd.	—

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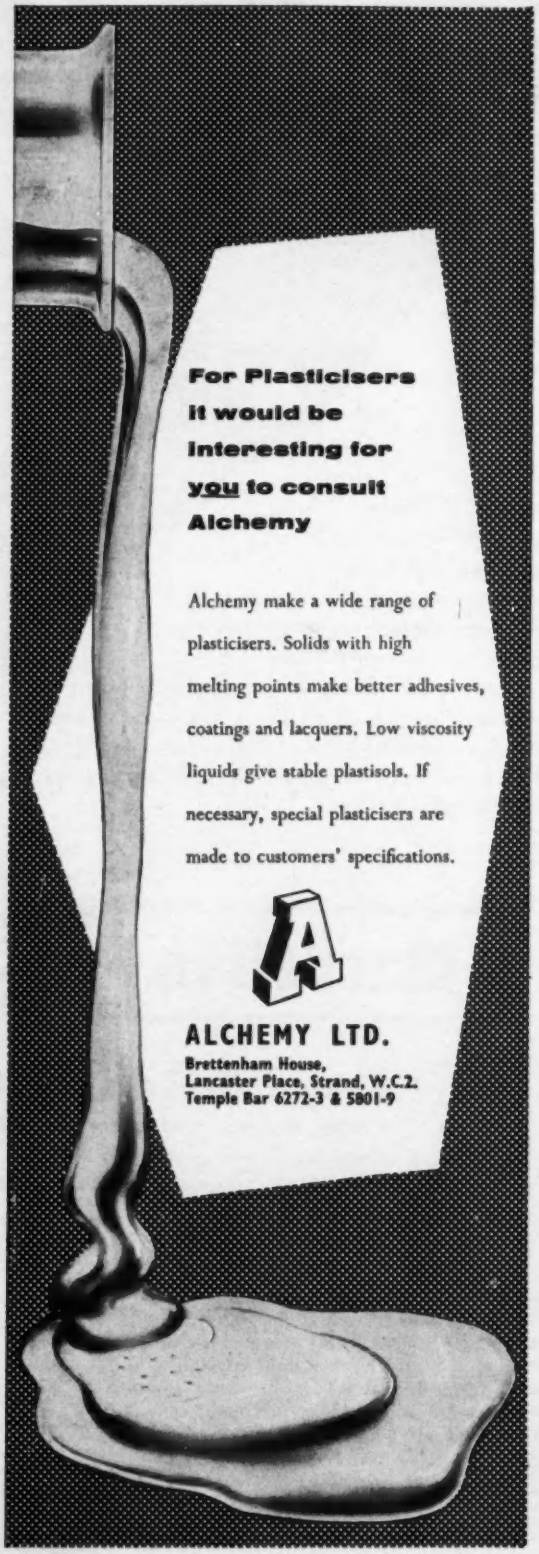

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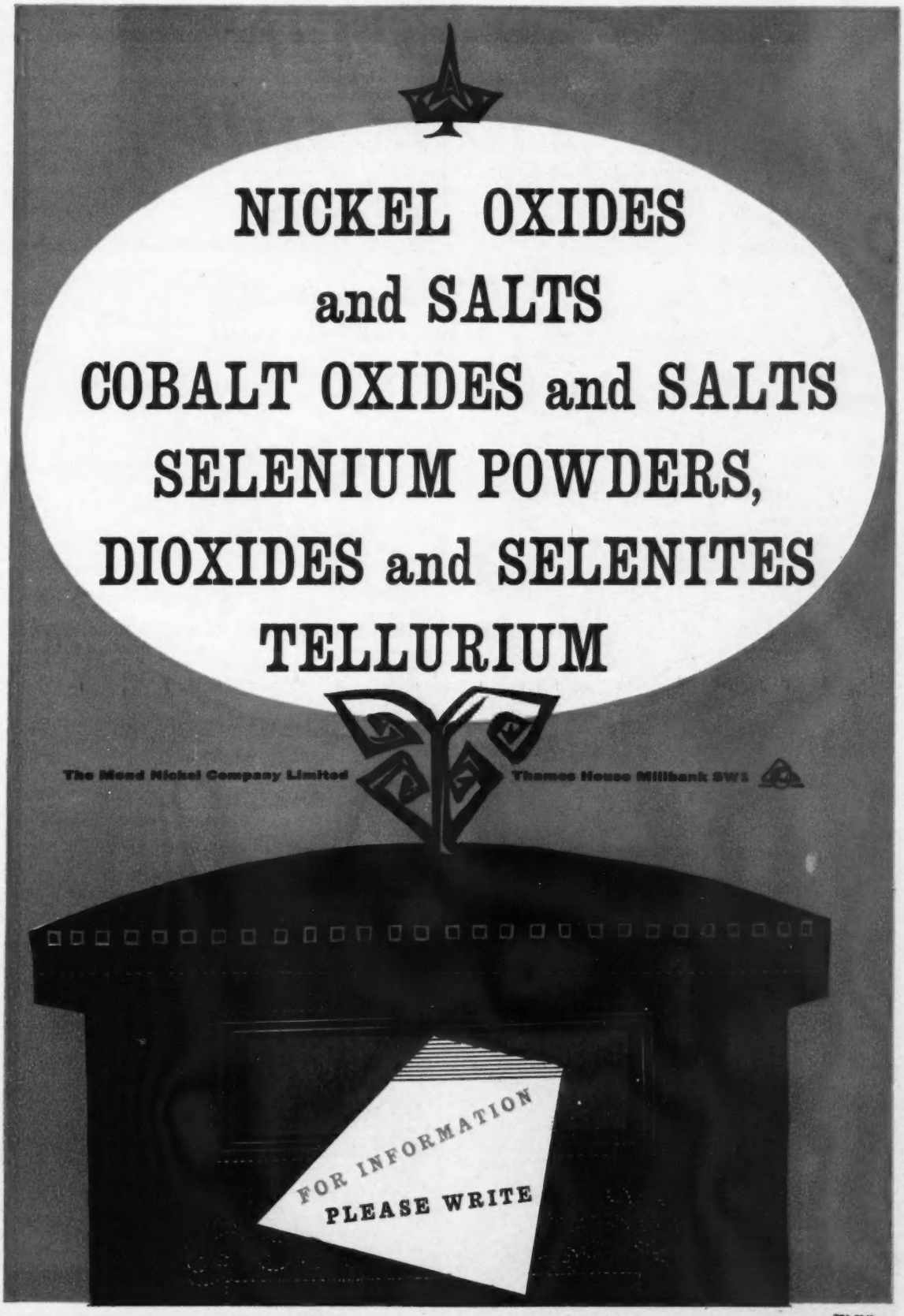
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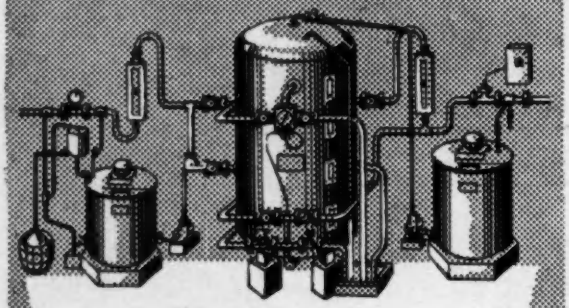

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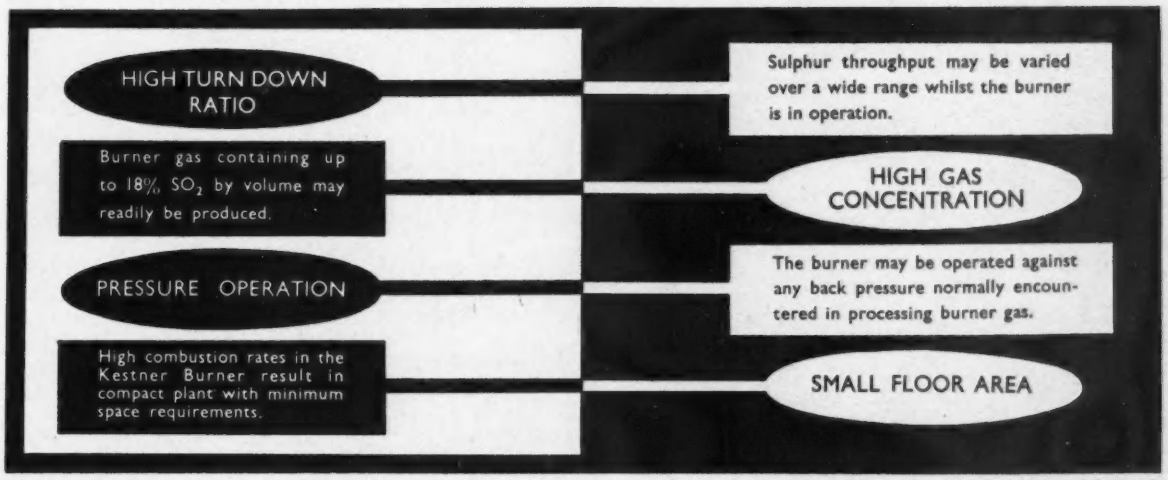
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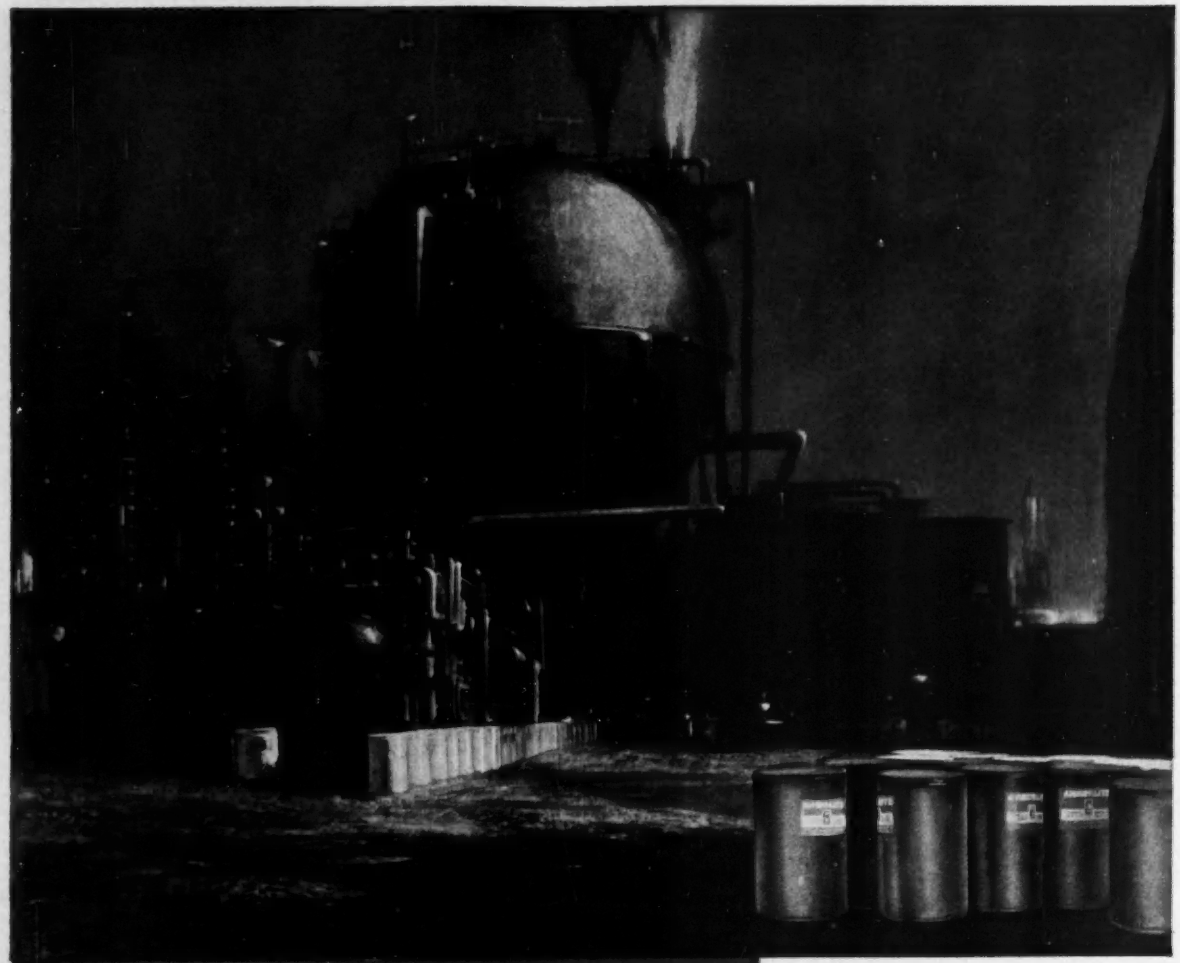

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AP13

VOL. 81 No. 2080
MAY 23 1959

CHEMICAL AGE

Telephone: FLEet Street 3212 (26 lines)
Telegrams: Allangas - Fleet - London

BOUVERIE HOUSE · 154 FLEET STREET · LONDON · EC4

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IN THIS ISSUE

Treating Sour Gasoline	854
British Titan's TiO_2 Extensions	855
In Parliament	855
Distillates	856
Synthesis of Organotin Compounds	857
Hickson's Indian Dyestuffs Plant	858
Hüls' Advances in Plastic Production	859
Low Temperature Distillation of H Isotopes	861
Cold Probe Corrosion Test	862
O.C.C.A. Polymer Conference—3	863
Analysis of Tar Acids—2	865
Triethylenediamine as One-Shot Catalyst	866
Overseas News	867
Chemicals at Hanover Fair	868
People in the News	869
Commercial News	870
Market Reports	870
Trade Notes	872
Diary Dates	872
Government and Water Research	873
Patents	874

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WATER RESEARCH

INCREASING demands of industry for water, the depletion of underground sources, the increasing costs of trunk mains from distant sources and the need to turn to polluted rivers for raw water, are all contributing to the urgency of scientific research into water problems.

In a report from the Central Advisory Water Committee published as a White Paper, for instance, it was estimated that water demands in England and Wales will increase by 23% in the period 1955-65. This means that the 3,600 million gallons a day gross in 1955, will rise to about 4,500 million gallons by 1965.

Recent surveys have indicated that in large urban areas supplies have increased by 42% during the period 1938-56 and in the last eight years by 12½%. Of the total usage of water only some 19% is used domestically, and of the remainder about 45% is taken by industry and a similar percentage by agriculture and irrigation.

For some years now informed circles in the U.K. have suggested that a more intensive study of the whole water economy should not be long delayed. Despite heavy capitalisation (to the tune of about £700 million) the water industry had no up-to-date research establishment when there was a vast field of work open to research by D.S.I.R., the Water Research Association (W.R.A.) and undertakings' own water engineers and chemists.

Many of the present-day problems associated with water supplies can only be solved by research and the W.R.A. since its formation over three years ago has been struggling to cope with the problems so far as its finances would allow. In fact fundamental studies have been impossible and although such knowledge is known to be a pressing and essential need in the water industry, the W.R.A.'s limited resources have made it impossible to undertake investigations of this nature in a complete and speedy manner. However, as Sir George McNaughton pointed out at the recent extraordinary general meeting of the association (see page 873) while work properly falling to the W.R.A. has to some extent been prevented, it has done most useful work in its short life.

Government aid to the association has increased to over £20,000 a year, but it has been decided that as research needs of the industry are now so pressing, the annual subscription is to be doubled, so that a £27,000 Government 'Grant in Aid' can be 'earned'. D.S.I.R., with the need for further research strongly in mind, is offering a grant of £15,000 for five years, provided membership income is increased to £30,000 a year. In addition they offer £80 for every £100 membership income over £30,000, thereby making a maximum possible grant of £27,000 a year.

To earn this grant the industry has to provide some £45,000 a year at least. Sir George estimated that the annual income of the industry must be about £50 million a year and this would mean that total contribution to research would, on the average be less than 0.2d per £1 of revenue. It is understood that the average investment in research of modern industrial companies in Great Britain is of the order of 2¼d in the £1, while the more successful companies have found it more to their advantage to finance research in their special fields to a much greater extent.

How the necessary money for W.R.A. will be raised is not certain, but research work in the fields of water conservation, treatment and distribution is of vital importance, particularly to the chemical industry.

Speaking on behalf of D.S.I.R., Dr. B. K. Blount points out that in research associations there is co-operation between the different parts of industry and Government. If more research is needed then it is sensible to do it co-operatively since both money and manpower can be saved. That the D.S.I.R. should be the Government department concerned with W.R.A. is very suitable. D.S.I.R. already has considerable interest in water as in the work of the Water Pollution Laboratory and the Hydraulics Research Station through the hydrodynamic studies carried out by the Mechanical Engineering Research Laboratories and through the British Hydromechanics Research Association.

The W.R.A. is controlled by its Council and D.S.I.R. has nominated three members only to sit on it, but the association as such is completely autonomous. The two 'visitors' nominated, Professor F. H. Garner, director of Birmingham University's chemical engineering department, and Mr. C. S. Tanner, of I.C.I., have the duty to report in confidence to the Research Council of the department on the work and progress of the association, but also can be regarded as advisors.

The Government's terms to the association indicate its very definite approval of the work that has been done up to date in bringing the W.R.A. into being. It also signifies, as Dr. Blount confirmed, the Government's agreement with the programme outlined for the next few years and especially the move to Buckinghamshire, and finally it undoubtedly recognises the value of research in this subject and the value to the Government.

Among problems likely to be on W.R.A.'s programme is that of re-use of water. Re-use of water used for conveyance of heat in cooling and heating systems is common practice, but collection and return of condensate from steam generations is considered to leave much to be desired. With water shortage growing, the less desirable sources such as sewage effluents and brackish water which are capable of being treated, will have to be considered for use.

Methods available for keeping the so-called 'water conveyor' usable are settling and filtration processes, precipitation processes, biological and ion-exchange processes.

STRENGTH OF CERAMICS

AS part of a programme to develop a better understanding of the factors controlling the strength of ceramic materials at high temperatures, the National Bureau of Standards is investigating the strength of aluminium oxide single crystals (sapphire and ruby). J. B. Wachtman, Jr. and L. H. Maxwell of the Bureau staff have been using temperatures between 600° and 1,000°C. The study supports the theory that, in properly oriented crystals, plastic deformation relieves stress concentrations, thus increasing crystal strength.

Widespread use of ceramic materials such as aluminium oxide in electrical insulators and in aerodynamics makes it desirable to investigate the factors controlling their strength at elevated temperatures. Study of pure polycrystalline ceramics is, however, complicated by grain boundaries and by interaction of the single crystal grains. To eliminate these complicating factors, properties of single crystals and their effects on polycrystalline materials have been studied.

The strength of polycrystalline aluminium oxide is known to decrease with increasing temperature above 800°C, but, in one set of measurements on single-crystal aluminium oxide, crystal strength increased with temperature in this range. It was suggested that this might be caused by very small amounts of plastic deformation, that is, failure may have begun at points where the local stress was much greater than the average applied stress. Plastic deformation might then have reduced the degree of stress concentration with the result that a larger average stress could be sustained before failure occurred.

To test this theory, both sapphire and ruby crystals were favourably oriented (45°) for plastic deformation, and modulus of rupture was measured on the crystals as a function of temperature up to 1,000°C. For comparison, like studies were made on unfavourably oriented (0°) sapphire. Apparatus used for the modulus-of-rupture measurements was recently developed at the Bureau in similar studies on uranium and thorium oxides. It permits measurements to be made in a vacuum, or in an inert or oxidising atmosphere at temperatures up to 1,200°C with a controlled loading rate.

Results obtained are consistent with the view that microscopic plastic deformation at stress concentrations is responsible for the rise in strength, which actually begins above 600°C for the single crystals. Supporting this assertion are the large increase in strength for both 45° sapphire and 45° ruby and the large scatter in strength values obtained at temperatures below 600°C as compared with those at higher temperatures. It is suggested that partial relief of stress concentrations should decrease the scatter in strength values.

Decrease in strength of polycrystalline aluminium oxide above 800°C, which is in contrast to results for the single crystal, was thought to result from stress concentrations caused by grain boundary slip. Support for this idea was obtained by measuring Young's modulus and the internal friction as a function of temperature for the polycrystalline material. Both measurements indicated stress relaxation characteristic of grain boundary slip.

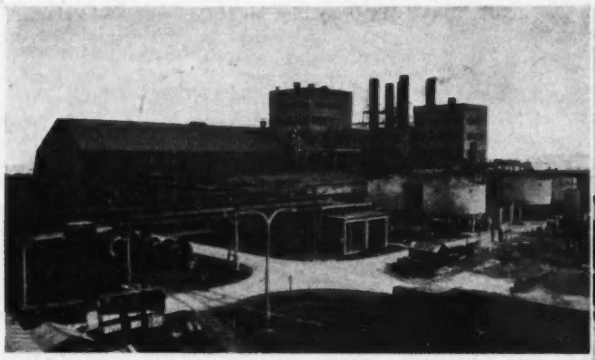
TREATING SOUR GASOLINE

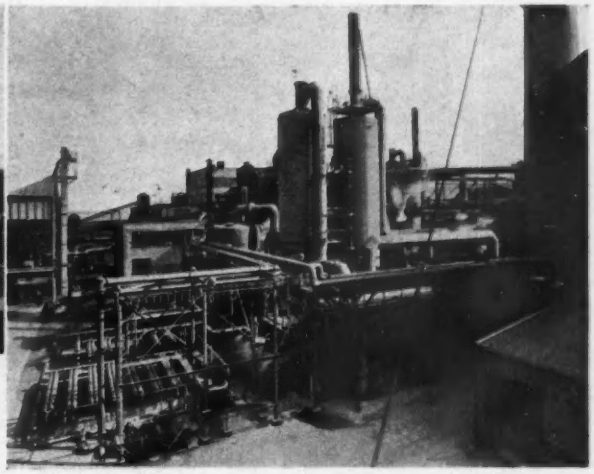
PROCESS patent U.S. No. 2,862,804 recently issued to C. O. Petty, La Gloria Oil and Gas Co., La Gloria, U.S., and detailed at the recent American Chemical Society's Boston meeting, discloses a completely new process for treating sour gasoline. La Gloria, it is revealed, have been treating gasoline with ethylene oxide as 'sweetening' agent on a 3,000 barrel/day scale at their Texas refinery for over three years.

In the Petty process objectionable mercaptans in the gasoline are treated with ethylene oxide. Hydrogen of the mercaptan joins the oxygen of the ethylene oxide to give an alcohol with a sulphide linkage in the middle. Reaction is stated to be slow at ordinary temperatures without a catalyst. An alkaline catalyst is helpful, but addition of phenols to the alkaline solution speeds up the reaction so that mercaptan removal is almost complete after five minutes contact time.

The unit used at La Gloria's Tyler, Texas, plant is a simple vertical packed tower through which a phenolic caustic solution circulates continuously. Gasoline flows upwards through the tower, countercurrent to the aqueous phase. Liquid ethylene oxide is metered into the gasoline just before the scrubber unit is reacted. Operation and maintenance costs are claimed to be relatively low, process costs being mainly related to consumption of chemicals. One barrel of light catalytic gasoline is stated to take an average 0.35 cents' worth of caustic soda and 0.60 cents' worth of ethylene oxide. For a thermal gasoline the costs are 1.30 cents and 2.25 cents respectively. Phenol consumption is said to be practically nil.

Research chemists of Sun Oil of the U.S. reported another process for treating sour gasoline (U.S. patent No. 2,850,433). This involves air oxidation of mercaptans to disulphides while the gasoline is percolating through a bed of high-iron bauxite. Suggested cost by Sun Oil on streams such as straight-run or thermal gasolines, is about 0.6 cents/barrel as against about 5 cents/barrel for caustic treatment of such stocks.





A general view of the new developments of the Grimsby titanium pigment producing works of British Titan Products Co. Ltd. is given in the left-hand picture. On the right is the new acid plant

70,000 Tons a Year Capacity at British Titan's Extended TiO_2 Plant

COMPLETION of a major extension to the titanium pigment plant of British Titan Products Co. Ltd. at Grimsby brings the total output of the Grimsby plant to 70,000 tons a year.

The extension has taken two and half years to build and has cost over £2 million. It is concerned mainly with the manufacture of the various rutile grades of titanium pigments, supplementing the production of anatase grades at the B.T.P. plant at Billingham.

New wagon-tippling plant and an automatic conveyor system feed a new ilmenite silo, with a capacity of 20,000 tons, loaded from the top. Ilmenite preparation plant including oil-fired drying and ball-mill grinding machinery, and a digester unit with an output of 50 tons TiO_2 per day has been erected. To cope with this capacity, a sulphuric acid plant capable of producing 180 tons a day has been included in the unit, with ancillary power sub-stations and air compressors. The plant is fully instrumented, production processes being regulated from centrally positioned control-rooms.

The titanium liquor produced by the new extension plant is pumped under pressure for refining and final stages of production at the main British Titan plant. The present stage of new building is completed with a large raw materials store.

In Parliament

No Methane Investment Until Trial Results Assessed

GAS Council's investment in the liquid methane tanker has been limited by the Minister of Power to the needs of the experiment. The Minister did not intend to consider any application to increase their investment until he had received a full technical and economic assessment of the results of the trial shipments.

This was stated by Sir Ian Horobin, Parliamentary Secretary, Ministry of Power, in the House last week. He added that it was not yet possible to assess the cost of producing town gas from liquid methane. Average London cost of production from coal and oil was roughly 16d and 14d per therm, respectively.

£5 Gasification Plant Will First Gasify Oil

Since nationalisation, spending on research into coal gasification has amounted to £1.25 million. In addition the Gas Council has undertaken construction of a new type of high pressure gasification plant at an estimated cost of some £5 million. In order to solve chemical engineering problems, the plant would be used initially to gasify oil, but in its final development it was planned to gasify small coal. This reply was given to a written question by Sir Ian Horobin, Parliamentary Secretary, Ministry of Power.

Antibiotics are Not Authorised for Use in Cosmetics

So far as the Government were aware, no cosmetic preparations including antibiotics were on sale in this country. They were assured that no such preparations were manufactured here, and none were known to be imported. The scope for the uncontrolled sale of such preparations was in any case extremely limited, as the sale or supply of most antibiotics was controlled under Part II of the Therapeutic Substances Act, 1956. This was stated in the House of Lords last week by the Earl of Onslow.

No proposals had been made to the Ministers to relax the requirements of the Act so as to permit the free sale of cosmetic preparations containing antibiotics. The Government were satisfied that the statutory provisions were sufficient to avoid any risk to health from the sale of cosmetic preparations containing antibiotics. Anyone found selling those things without proper right or control was liable to a fine of £100 and, on subsequent convictions, to imprisonment.

Fertiliser Subsidy 1958-59

Latest estimates of subsidies paid during 1958-59 include £26 million under the general fertilisers subsidy and £9.2 million under the lime subsidy. These figures were given in the House last week by Mr. John Hare, Minister of Agriculture, Fisheries and Food.

Humglas' New Contract

HUMPHREYS AND GLASGOW LTD., London, have obtained another contract for the supply of gas plant to Japan, their third in recent months and the ninth in all.

The latest is for the installation of two units of carburetted water-gas plant to be erected at Kanazawa.

The contract, which has been placed by the Okura Trading Co. Ltd. for the Kanazawa Gas Co., also covers a waste heat boiler and ancillary equipment.

Production of gas from each of the two units will be 400,000 cu. ft. a day. Value of the contract is about £25,000.

Useful Application of Tin Dithiol

Attention of the Tin Research Institute has been drawn by Dr. R. E. D. Clark to a property of dithiol which, it is believed may have some useful applications. Tin dithiol is formed, he reports, as a scarlet precipitate when a dithiol solution is added to a tin salt at a suitable acidity. The scarlet tin dithiol, however, changes its colour when it is heated. Just below 100°C, a change towards purple is noticeable and before 200°C is reached the colour is a deep purple or black. On cooling the original scarlet colour returns immediately. Dr. Clarke therefore suggests that tin dithiol, incorporated in a suitable paint vehicle, might be a useful temperature indicator.

DISTILLATES

★ ONE of the most striking aspects of the Chemische Werke Hüls works, which covers an area of approximately 2,500 acres, is its regular lay-out. This I learn is due to the fact that the whole undertaking was planned on the drawing-board and did not grow up haphazardly in the course of years, as so many other chemical works have done.

Some 12½ miles of roads, raised about 10 ft. above ground level owing to the high water level, divide the area. The roads, which run at right angles, are numbered and so give a clear idea of the lay-out. Bridges total some 7½ miles and carry some 370 miles of pipeline. There is also some 25 miles of railway tracks laid in one direction, from east to west only, thus avoiding crossings.

Almost in the centre of the works area is the old power station with an installed capacity of 204,000 kW; this is sited some 1,500 ft. from a coal mine. The company's new power station is one of the most up to date in Europe. It supplies energy to the Bunawerke Hüls G.m.b.H. For economic reasons, Hüls find it preferable to sell the power produced during daytime hours and to buy power at night.

The works area has been laid out in accordance with purely rational considerations. Thus all those installations which use a great deal of steam, such as the distillation plants, which number about 200, were erected south of the old power station.

★ I WAS interested to learn recently that Esso Petroleum expect to save about $2 million through the use of aluminium air-fin coolers at their 100,000 barrel refinery now being built at Milford Haven. In another refinery being built at Whitegate, Eire, Esso and others expect to save $1,200,000 a year by using similar equipment.

The savings are realised through the reduction or elimination of cooling water and effluent systems. This new technique was used at Esso's Fawley petrochemical facilities and in 'Distillates', 13 December last, I said:

"For the first time in a U.K. oil refinery, air coolers are used on a large scale. Supplied by Head Wrightson, these form part of the butadiene dehydrogenation unit and represent a big step forward in the prevention of water pollution".

★ FIRST opportunity for Western chemists to assess antibiotic developments in East Europe is being provided this week at an international symposium in Prague. Papers are centred on the fundamental and practical aspects of antibiotic production.

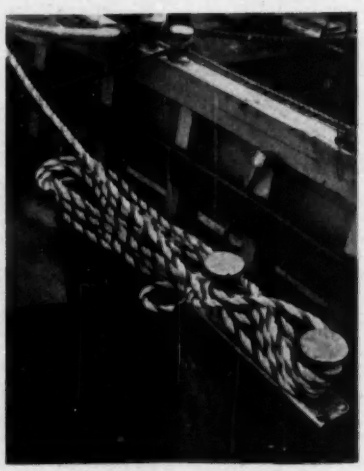
I learn that details of several antibiotics "hitherto unknown in the West, some of which appear to be active against experimentally-induced tumours while others possess antifungal or antiviral properties" are to be given. Delegates will discuss the treatment of plants and fruit diseases; preservation of meat and fish with trace quantities of tetracycline; growth-promotion when tetracycline is added to normal diet; the effect of botrymycin on the quality of Tokay wines.

In technological sessions, discussions are covering the influence on output changes in media, constituents and fermenting conditions. The adverse effect on streptomycin production of trace quantities of iron and means of overcoming it will be discussed by several authors. In all there will be 200 papers, mostly from East European delegates, with five from the U.K. and eight from the U.S. Distinguished contributors include Nobel prize winner Professor E. B. Chain, director of the International Centre for Chemical Microbiology, Rome, and Dr. Henry Welch, chief of the U.S. Food and Drug Administration's Antibiotic Division.

★ IT SEEMS that CHEMICAL AGE'S exclusive report of the annual report of Dr. Carlo Faina, president of Montecatini ('Overseas News', 9 May), may have given the impression that the company's nitrogen plants were operating at 60% of their capacity. In a letter to the editor, Dr. Faina points out that he was referring to Italian capacity in general and not to his own company's facilities.

Dr. Faina says that in 1958 Montecatini's nitrogen plants operated at their maximum capacity and that the company is certain that this performance will be repeated during the current year.

★ ANOTHER new and potentially important use for polythene is in the form of high tenacity yarn for rope and cordage manufacture. This new monofilament, known as Courlene X3 has been developed over three years and is now in full production at the Coventry Works of British Celanese. It is spun from Rigidex high density material supplied by British Resin Products.



Several important advantages are claimed for the new yarn. First its low specific gravity (0.96) produces ropes of extremely light weight which have a strength comparable with nylon and Terylene, as well as good abrasion resistance. These properties are maintained over a wide temperature range, from −70°C to 100°C. The ropes are unaffected by chemicals, oils and greases and may be used for many purposes, including mooring and running tackle.

I am told that ropes made with Courlene X3 do not absorb water and are thus unaffected even by prolonged immersion; they do not swell, stiffen or rot. Sailors —professional or amateur—will appreciate the fact that the low density of the yarn means that ropes float in both fresh and salt water, also the fact that there appears to be no tendency to slip through the fingers when wet.

★ EIGHTEEN months ago CHEMICAL AGE criticised U.K. pharmaceutical firms for the paucity of their spending on research (C.A., 1958, **79**, p. 910). Last week the Hinchcliffe Committee said much the same. The £4 million a year currently being spent by the industry in this country compares with a U.S. total of about $170 million and a Swiss figure of £7 million a year.

Three-quarters of U.K. spending on pharmaceutical research is made by three firms. In March 1957, we spoke of the danger of buying research from the U.S. and the Continent. Although there have been some notable British achievements in this field in recent years, there has not been the steady flow of discoveries and new products, originally conceived, which could be expected from the industry.

In some quarters the small research budget is said to reflect the Government's parsimonious treatment of the Medical Research Council, which receives only £4 million a year. That may be, but, there is no doubt that one of the main reasons is the domination of production facilities in this country by companies owned by U.S. or Continental parent companies. Their policies generally are not to repeat basic research that is carried out in their base countries; laboratories of British subsidiaries tend to concentrate on processing research, and technical service.

★ "THE only guide books that tell you anything"—this was the verdict on the Blue Guides pronounced by a travel agent at a recent meeting of representatives of the tourist industry.

At all events, the new edition of Muirhead's *Blue Guide to Scotland*, published on 6 May, is devoid of padding and is, like all the volumes in the series, an invaluable and money-saving companion to any traveller in the country concerned.

Alembic

INDUSTRIAL SYNTHESIS OF ORGANOTIN COMPOUNDS

ONLY three methods of making the tin-carbon bond, which are of industrial interest, have been discovered. These are: (1) direct reduction of halogenated hydrocarbons with metallic tin or its alloys; (2) a Wurtz reaction and (3) a Grignard reaction. These three methods of synthesis are discussed by Dr. C. R. Cramer in the Tin Research Institute's latest issue of *Tin and Its Uses* (Spring, 1959, No. 46, p. 7).

Throughout his paper Dr. Cramer uses 'butyl' as the typical alkyl, and 'phenyl' as the typical aryl group, although other alkyl or aryl groups are, or could be, used.

Starting materials used today in the preparation of organotin compounds, are tetrabutyltin and the oxides and chlorides of tributyl, dibutyltin and triphenyltin. The choice of the butyl group is stated to be justified both scientifically and economically, but at the same time Dr. Cramer suggests that other types such as octyls, vinyls, allyls, benzyls, cyclohexyls or tetrahydrofurfuryls could replace a part of the butyls, and industry should therefore bear this in mind and adopt flexible procedures.

Methyl Chloride

Direct synthesis. The simple reaction

$$2CH_3Cl + Sn \rightarrow (CH_3)_2SnCl_2$$

is only possible with methyl chloride; higher alkyls react only in insignificant amounts. Also van der Kerk *et al.'s* method of reacting ethyl chloride or bromide with this magnesium alloy (Mg_2Sn) is not applicable to butyl chloride. Using other alloys of tin—e.g. aluminium, zinc, beryllium and sodium, tin has been combined with vinyl and allyl groups and even with butyl, but the yields are not high and early industrial application is not expected.

Indirect methods. Indirect routes have been found to give the best results; the Wurtz reaction by which the 4 valencies of the tin atom can be progressively substituted and the Grignard reaction of an organomagnesium chloride with tin tetrachloride. In both cases stannous chloride is the starting material.

The Wurtz reaction. In 1947, a patent described the reaction of chlorobenzene and tin tetrachloride with metallic sodium. Under suitable conditions of temperature, time, type of solvent, proportion of reagents, form of sodium, etc., a mixture of organotin compounds (R_4Sn, R_3SnCl, R_2SnCl_2) is formed in relatively high yields.

With the idea of substituting for the stannic chloride an organotin such as dialkyltin dichloride, van der Kerk's group have succeeded in devising a method applicable to industrial production by combining Wurtz and Koecheskoff reactions.

Troublesome reduction of tin tetrachloride is eliminated and formation of other secondary products of the reaction can be held low. Also hexabutyl distannate can be reconverted into tributyl tin chloride by simple treatment with chlorine. It is unnecessary to push alkylation to the end of the Wurtz side and it is sufficient to attain a value of 3.6 to 3.8 instead of 4 alkyls. Reaction time is shortened and reducing effects minimised.

Using the Koecheskoff reaction, only sufficient tetrachloride of tin is added to provide an alkylation figure above 2 (e.g. 2.2) to avoid all formation of butyl-tin trichloride. With an index of 2.2, tetrabultyl tin is completely transformed and the reaction product contains dibutyl tin with a small percentage of tributyl tin chloride.

With the demand for a minimum purity of 99%, it is necessary to distil the product after the Wurtz-stage.

The Grignard Method. Using tin tetrachloride and appropriate organo magnesium compounds, tin alkyls, alkenyls, cycloalkyls, aryls and heterocyclic compounds can be produced simply and without side reactions. On an industrial scale, however, the reaction is dangerous due to the large quantities of ethyl ether involved and therefore has to be carefully controlled. The problems involved are:

(1) That reaction should start easily without delay. This involves use of an active and reliable initiating substance.

(2) Reaction must proceed vigorously until reactants are exhausted. Structure, size and quality of the magnesium turnings are important as are the initiator, the type, quantity, and purity of the solvent and the means of stirring.

(3) The solvent should stabilise the reaction system. A chlorinated hydrocarbon should be included in the most volatile constituents so that, if a rise in temperature occurs, this reactant volatilises, thereby reducing the speed of the reaction and conversely if the temperature falls, the hydrocarbon condenses and so increases the speed of reaction.

(4) Cooling has to be controlled. It is possible to operate on 5 m.³ with this reaction, and higher ethers such as tetrahydro furane, cyclohexane, octane, isooctane, etc. The Grignard reaction, according to Dr. Cramer, can now be carried out in the presence of tin chloride, combining the separate steps of the synthesis into one cycle only. A similar cycle worked out by Professor van der Kerk for the Wurtz reaction is the subject of a patent (Brit. Pat. 786,545). Experience has shown that it is possible to obtain regularly very high yields with conversion, e.g., of 97.5% of stannic chloride, 88 to 92% of butyl chloride and 85 to 88% of magnesium.

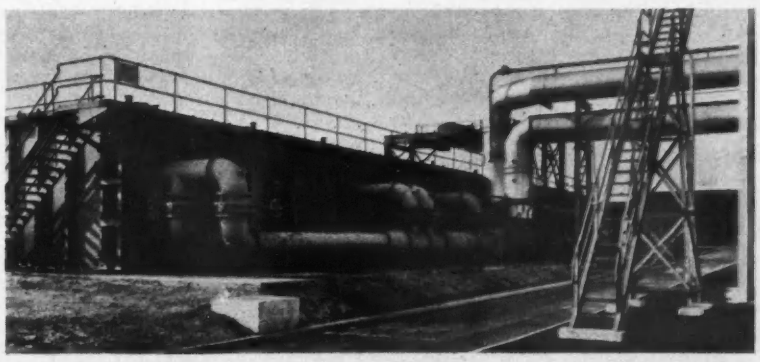
Advantages of the Grignard method are stated by Dr. Cramer to be: excellent yield; pure products; low risk; adequate scale (up to 1,000 kg. at a time in a 5 m.³ reactor); minimum of supervision; negligible corrosion; great flexibility in production. Disadvantages are: use of expensive raw materials ($SnCl_4$ and Mg); high cost of installation; bulky equipment; large quantities of solvents which have to be recovered, purified and dried after each cycle.

Economic comparisons. A utilisation of 94.6% of the cost of materials is estimated for the Grignard reaction. With the Wurtz method using sodium, the equivalent figure is 92.5%. On the basis of the theoretical synthesis

$$2\ BuCl + Sn \text{ (granulated with catalyst)} \rightarrow Bu_2SnCl_2$$

the equivalent utilisation is 72.5%. (Polkinhorne and Tapley, British Patent 761,357, give the figure as 84%.)

North Thames Gas Board's Deep Box Purifiers at Romford's Gas Re-forming Plant



Deep-box purifiers at the North Thames Gas Board's Romford gas re-forming plant, which was opened by Lord Mills, Minister of Power, on 1 May. Gas for the plant is purified before it leaves the Thames estuary oil refineries, but a small amount of sulphur is converted to hydrogen sulphide in the re-forming process, making further purification necessary. Iron oxide is used to remove the hydrogen sulphide

POLYETHYLENE GLYCOL A NEW MEDIUM FOR RESIN FLUX

THREE polymers of ethylene glycol, having nominal molecular weights of 200, 550 and 4,000 respectively (obtainable from Shell Chemical Co. Ltd.) have been examined as alternatives to ethyl alcohol in soldering fluxes by W. R. Lewis (*Tin and Its Uses, Spring*, 1959, No. 46).

The '200' polyethylene glycol is described as a fairly free-flowing liquid at ordinary temperatures, with a consistency similar to ordinary gum mucilage; the '500" polymer is a soft white paste, which melts on a water bath; and the '4,000' polymer is a white flaky substance which melts readily. All three take up 20% by weight of water-white resin, Poly-pale (Hercules Powder Co.). Solder spreading tests have shown, states Lewis, that these resin carriers are practically indistinguishable from ordinary resin in industrial alcohol of the same concentration.

Main advantage of these polymers as resin flux media is found to be in their much lower volatility than alcohol. This property is particularly of value in avoiding evaporation of solvent from resinous soldering fluxes when alcohol is used near solder dipping-baths or other hot places. Experiments have shown that after four hours on the water-bath, the evaporation loss from 100 gm. of 20% resin in polyethylene glycol of the '200' polymer, was only 2 gm. The '550' lost 1 gm. in the same time and the loss on the '4000' polymer was negligible.

There is no fire risk with the polyethylene glycols since their flash-points are high:

'200'	flash point	171°C.
'550'	" "	238°C
'4 000'	" "	271°C

All three polymers have been found to readily dissolve 'activated' resin containing an amine hydrochloride and activity has remained unchanged compared with ethyl alcohol solution of the same activated resin. Minor advantages which may follow from the use of the polymers as resin flux media listed by Lewis are no unpleasant odour and no risk of intoxication from fumes. During soldering tests on copper, no unpleasant odour or charred residues resulted.

Monographs for Teachers

The first two items in a new series of monographs for teachers have been published by the Royal Institute of Chemistry. These are entitled: 'Principles of Electrolysis' by C. W. Davies, and 'Principles of Oxidation and Reduction' by A. G. Sharpe.

This series aims at presenting concise and authoritative accounts of selected well-defined topics in chemistry for the guidance of those who teach the subject at G.C.E. Advanced level and above. Copies may be obtained from the Education Officer, 30 Russell Square, London W.C.1, price 3s 6d each.

Indo-British Dyestuffs Plant Opened

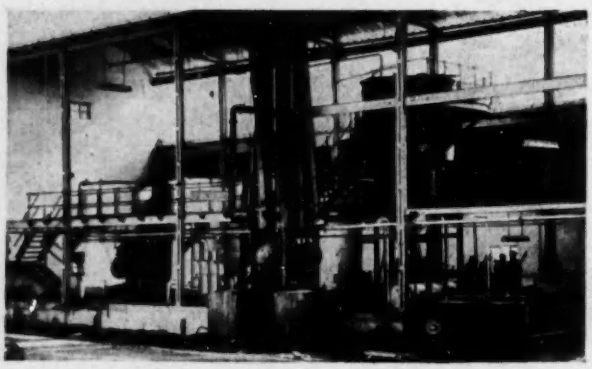
HICKSON and Dadajee Private's dyestuffs plant on the outskirts of Bombay, was opened on 2 May, less than a year after work started on the site.

Initially it is manufacturing sulphur black and Photine optical whitening agents, but a licence has been obtained from the Indian Government for the manufacture of 21 intermediates for dyestuffs, paints and pharmaceuticals.

British visitors attending the opening ceremony were Mr. Bernard Hickson, chairman, and Mr. G. K. Day, joint managing director of Hickson and Welch Ltd. Hickson and Dadajee Private Ltd. was incorporated in 1953 by Hickson and Welch Ltd., Castleford, Yorkshire, and Dadajee Dhackjee and Co. Private Ltd., Bombay. In June 1954, a temporary plant was erected in a leased factory at Sewree, and the manufacture of sulphur black began one year later.

Half of the output is now in liquid form.

Speaking at the opening ceremony, Mr. S. J. Wankhede, Minister of Planning, said home production had already reduced India's imports of dyestuffs from Rs.14 crores (£10.5 million) to Rs.11 crores (£8.25 million).



A view of part of the Hickson and Dadajee sulphur black plant on the outskirts of Bombay

Belfast Meeting on Polarography in June

A JOINT meeting on polarography will be held by the Polarographic Society and the Scottish Section, Society for Analytical Chemistry, at Queen's University, Belfast, on 26 June. Papers will be presented between 10 a.m. and 12.30 p.m. and 5 p.m. and 7 p.m.

Those intending to be present should notify Mr. J. Brooks, Analytical Research Section, I.C.I. Nobel Division, Stevenston, Ayr, not later than 31 May, stating whether they would like hostel accommodation.

Papers will deal with copper complexes in non-aqueous solution; rhenium and technetium; tissue respiration; a potentiometric method for acid-base titrations in acetone-water solutions; determination of nitroglycerine; fused salts; and polarographic estimation of mercury fulminate. Authors will be Dr. Z. Zigorski, Dr. R. J. Magee, Dr. I. S. Longmuir, G. F. Reynolds, A. F. Williams, R. L. Faircloth, and J. Hetman respectively.

Reusable Envelope from I.C.I.'s Hydex

ICI's Hydex pvc-coated synthetic material is used for the Envopak, a constantly reusable envelope developed by Envopak Ltd., Fulham Road, London, S.W.6, and shown here

Baker Perkins and T.B.E. Agreement

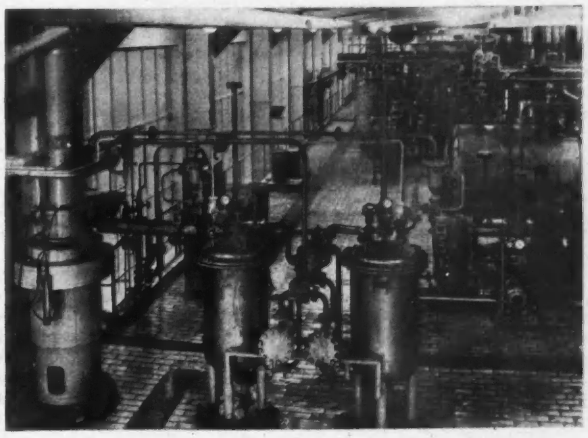
Baker Perkins have signed a preliminary agreement with the Israeli Ministry of Development and Israel Mining Industries for the exploitation of a process for mineral ore dressing and coal preparation based on the use of tetrabromethane.

Chemical Plant Deliveries, 1958

In a new regular series of statistics issued by the Board of Trade, it is stated that deliveries of chemical plant during 1958 were valued at £50.3 million. Quarterly totals for the year were: 1st quarter, £13 million; 2nd quarter, £13.2 million; 3rd quarter, £12.4 million; 4th quarter, £11.7 million.

Hüls' Advances in Plastics Production

Base Raw Materials Application—Tested



Pilot polymerisation plant for research and development work on high polymer at Chemische Werke Hüls in West Germany

PRODUCTION of Buna synthetic rubber and some of its by-products was the exclusive concern of Chemische Werke Hüls during the first years of their existence, but some 12 years ago steps were taken to pursue the development of plastics raw materials, raw materials for varnishes and detergents, and solvents. Today Hüls sell 600 different chemicals to all parts of the world.

Share capital of C.W.H. is shared by Chemieverwaltungs-AG., Frankfurt/Main (50%), Bergwerksgesellshalft Hiherina (25%), and Kohlener-vertungs GmbH. (25%). Turnover is largely based on plastics raw materials, the foremost plastics being polyvinyl chloride (p.v.c.) marketed under the trade name Vestolit. C.W.H's p.v.c. production capacity is likely to be among the largest in the Federal Republic. Polystyrene is also being marketed by the company under the trade name Vestyron. Here, too, Hüls have considerable production capacity, and it is noteworthy that by this summer Hüls is likely to become the largest producer of styrene on the Continent of Europe, with a capacity of 6,000 tons a month, as a result of the expansion of production installations for monomer styrene.

Ziegler Polythene Plant in Operation

Successful research carried out by the company on Ziegler low-pressure polythene has led to the construction of a polythene plant, with a monthly capacity of 500 tons. To the Hüls range of plastics materials is shortly to be added polypropylene, Vestolit P. Besides this range of plastics raw materials, Hüls today produces such interesting auxiliaries for the manufacture of plastics, as plasticisers, solvents and synthetic resins, detergents and ethylene oxides with their derivatives, chlorinated hydrocarbons, organic acids and industrially valuable gases.

Some 35% of the company's total turnover is accounted for by exports. The greater part—some three-quarters in fact—goes to the countries of the European market, although of late the ratio of European exports to overseas exports has shifted slightly in favour of the company's overseas customers.

'Heart' of the C.W.H. works is the electric arc plant which serves for the production of acetylene from gaseous hydrocarbons (waste gas from hydrogenation processes, coke-oven gas and natural gas, piped direct from Pentheim on the German/Dutch border and also brought in liquefied form by canal barges). At present the plant consists of 17 arc furnace units, a number of which have been converted to automatic operation.

Gaseous hydrocarbon, for the most part methane, enters a pipe 3½ in. diameter and 39 in. long. An electric flaming arc burns in this pipe, in the axis of which temperatures of 18,000°C. have been recorded. Average temperature in the water-jacketed pipe is 1,500°C., at which temperature conversion into acetylene and hydrogen takes place. Hydrogen is conveyed by long-distance pipelines to hydrogenation plants, fertiliser and detergent plants. A certain amount of carbon black is also formed. New type automatic measuring and remote control devices to improve production in the arc furnace units are employed and are incorporated in the plant's control panels.

Plastics production. The acetylene is used by Hüls for the manufacture of p.v.c. in combination with hydrochloric acid to produce vinyl chloride which is polymerised to emulsion and suspension forms of p.v.c.

The product of the emulsion process, which is in the form of a dispersion, passes through an atomiser which separates the solid from liquid. The atomising columns are conical in shape. The dispersion is introduced from the top by compressed air, and atomised through fine nozzles. A hot air current passes through the columns at the rate of 3½ million cu. ft. per hour. Water is evaporated off in the hot air current and solid p.v.c. powder drops to the bottom. It is then passed through large filler plants and through a dust separator to the storage lines.

In the granulating plant p.v.c. powder is mixed with stabilisers, dyes and, if necessary, with plasticisers. A noteworthy feature of this plant is the automatically-operated dosage meter which can be adjusted for every conceivable composition of the finished product, including the colouring, by means of a punched card. Changing of the composition means that it is only necessary to insert a different punched card. This same plant also processes low-pressure polythene. In order to make the material suitable for cutting, the plasticised compound is sprayed with cooling water. The water is then separated in reciprocating troughs and cyclones to obtain a dry material.

Raw materials used in the production of polystyrene are benzene and ethylene. The ethyl benzene produced is dehydrated to give the styrene monomer.

Polyester resin is also produced. It is composed of dicarborylic acid esterified with glycols. Styrene monomer is added and the polyester resin is produced in liquid form.

Low-pressure polythene is being made at Hüls using the Ziegler process, and chromium oxide and aluminium oxide as catalyst. Like all producers of Ziegler polythene. Hüls have had to resolve the problem of producing polythene free from catalyst. The plant is a large-scale one with a monthly capacity of 500 tons.

Polymerisation pilot plant. Hüls have a polymerisation pilot plant where runs from approximately 10 gall. up to 80 gall. are possible. Following flask polymerisation, the next step is polymerisation in 9 gall. reactors and in 80 gall. reactors. The plant is equipped with a large number of reactors made of a variety of materials in order to gain experience of the behaviour of the different materials which might later be used in building industrial plant.

Department for Application Techniques. In order to process the plastics raw materials produced at Hüls under industrial conditions, the company has built a miniature plant for the processing of plastics. The building houses the most widely used up-to-date processing machines for plastics, which are continually being replaced by new models. Plastics tubes can be drawn and cables insulated. Artificial leather cloth can be made as well as foils, and there is also a paper finishing machine. There is no lack of injection moulding machines of all kinds, nor of installations for deep-drawing of plastics sheets. One of the latest types of broad-slot nozzles producing such sheets was observed in operation. Centrifugal casting machines are being used to investigate the processing of

plastics. Suitable machinery has been installed for studying p.v.c. foam.

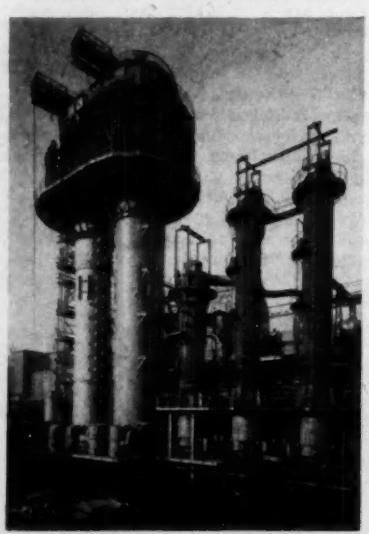
So successful are the techniques, etc., being employed in the development section, ostensibly to iron-out customers' problems and to allow Hüls to specify the properties of their plastics, that it is understood that new machines are being bought out of novelty goods produced and sold.

Plant for reclaiming sulphuric acid. A plant has been installed to process used sulphuric acid. It is largely automatic in operation. Residual sulphuric acid is dissociated to give sulphur dioxide at temperatures above 1,000°C, organic impurities being burnt to carbon dioxide and water. After passing through a scrubber, filter and drying plant the SO_2 is led to a contact furnace, where, with air, catalytic conversion to SO_3 takes place. The SO_3 so obtained is processed by conventional means to sulphuric acid. A control station attached to this plant features a graphic control panel, for temperatures, pressures, etc.

Trichloroethylene production. Trichloroethylene is recovered from acetylene and chlorine. A central control station also controls this plant which has its own miniature control panel.

Ethylene production. Ethane, propane, and to a limited extent, other gaseous hydrocarbons are converted to ethylene in a tube furnace or pipe still in which modern design principles have been incorporated. Production is again controlled from the central control station using automatic control instrumentation. The ethylene is used as raw material for production of ethylene oxide and styrene.

Acrylonitrile plant. At Hüls, acrylonitrile is produced at present in a large pilot plant, some 200 tons a month being made, using as basic raw materials acetylene and hydrogen cyanide. Production of hydrogen cyanide from methane and ammonia has been combined with production of acrylonitrile from acetylene and hydrogen cyanide, the entire plant being controlled from a single central control station. Because of the very complicated processes involved, it was not found possible to equip the central control station with a graphic panel. Control lights have been installed at individual positions, where temperature measurements are required or control instruments can be utilised.



New distillation tower for styrene at Chemische Werke Hüls, West Germany. This distillation is air-cooled

Ethylene oxide production. The most modern automatically-controlled production plant at Hüls is that for the manufacture of ethylene oxide, obtained from ethylene and air by the contact process. Some 35 different operations are automatically controlled, and the plant has a total of 281 warning lights, 186 recording instruments, and 229 indicating instruments in its central control station. From this section the temperature can be measured at 250 different positions throughout the plant. Six electronic compensating recorders are located in a single control panel; within 48 seconds each of them records 12 different temperatures.

Research at Hüls. Products which in 1950 were not part of C.W.H.'s production programme today account for more than 70% of the company's turnover of DM. 500 million (1958). Funds equivalent to 5% of C.W.H.'s turnover are appropriated for research purposes every year.

Of some 200 chemists and physicists employed at Hüls, more than half are exclusively concerned with problems of research and development. The other chemists and physicists distributed throughout the various divisions, also work on development problems at the plant sites.

A new research laboratory, built on skyscraper lines—it has eleven floors—contains special workshops and technical central laboratories. Advanced gas chromatographic apparatus is used for analysis and tests on high polymers and equipment is available for determination of the molecular weight of high polymers. A process has been developed to ascertain molecular weights which consists in determining the rate of sedimentation of the high molecular weight compounds in solution. A high-speed centrifuge is being used for this work, which is driven by an air turbine and which can reach a speed of 50,000 r.p.m. An acceleration of up to 200,000 times that of the earth can be obtained.

Other research under way at Hüls is in the production of acetylene by thermal processes without use of electrical energy, productions of olefins and diolefins. As a result of three years' research equipment has been designed which is effective in preventing progressive decomposition of acetylene in large diameter pipelines. This equipment has now been fitted into the plant's acetylene network.

Successful Operation at Bunawerke Hüls

Work on the new synthetic rubber plant, Bunawerke Hüls AG, in which Hüls has a 50% interest was completed last year and the plant has been in operation for about six months (see CHEMICAL AGE, 20 September, 1958, p. 481).

The plant which is producing 70,000 tons of buna a year, has a capacity of 90,000 to 95,000 tons. It consists of a butadiene plant using the Houdry process, a butadiene purification plant, a storage tank unit for intermediates, a polymerisation plant for production of rubber latex, a processing plant where the buna is coagulated, dried and baled, and a storage and dispatch shed. About 300 persons are employed in this plant. Petrochemical raw material, normal butane, is used as the basis of the process. This is supplied by Hüls in tanker lorries by the large refineries operated in Germany by Esso, BP and Gelsenberg Benzin A.G. It is then heated to a temperature of 600°C and subsequently passes into six reactors about 19 ft. in diameter and approximately 45 ft. long, and is dehydrated by means of a catalyst.

In the plant where the butadiene is purified and saturated, the gas mixture is combined with a solution of cuprammonium acetate which selectively extracts the butadiene from the mixture. After the final distillation the butadiene and the monomer styrene can be polymerised to produce synthetic rubber latex.

Polymerised material passes to 20 mixing drums with an internal ammonia vertical-tube evaporator system. This serves to cool the reaction which takes place at a temperature of 5°C and at atmospheric pressure. Apart from the two basic components, butadiene and styrene, polymerisation of the latex requires the addition of other chemicals, including an emulsifier, which through a complicated dosage meter are added to the process by automatic means. When the polymerisation reaches 60% the process is chemically interrupted, the monomers which have not yet been polymerised are reclaimed, and latex is pumped out to be coagulated, dried and baled.

Coagulation of synthetic rubber from latex is carried out with sulphuric acid. Rubber flakes then are washed neutral in open mixing drums. Filter presses then mechanically extract the water and the resulting cake, after being ground in a disc crusher, is pneumatically moved on to a plate conveyor for hot air drying, past automatic scales to the baling press. Automatically packed the 55 lb.-bales then reach the dispatch department from where they go to the rubber factory for further processing. This entire plant is automatically controlled, the whole process hinging on dosage meters.

Production of butadiene, on an acetylene basis, was reduced in 1958, since this material can be imported in the quantities required, and at favourable prices from Distillers Co. Ltd.'s British Hydrocarbon Chemicals plant and Italy. The acetylene supplies have thus been released to expand Hüls' business in solvents. No further expansion of the Houdry process plant is contemplated as the extra butadiene required will be bought. There is ground space, however, in the remainder of the synthetic rubber plant to duplicate all processes from polymerisation onwards.

L.T. Distillation of Hydrogen Isotopes May Give Bulk Quantities of Deuterium

EXPERIMENTAL work begun at the National Bureau of Standards' Cryogenic Engineering Laboratory (Boulder, Colorado) in 1955 has advanced a practical solution to the problem of separating hydrogen isotopes (1). A pilot plant capable of distilling liquid hydrogen by fractionation has yielded specific amounts of pure liquid deuterium and deuterium-free hydrogen.

The unit was designed by T. M. Flynn, K. D. Timmerhaus, and D. H. Weitzell of the laboratory and they suggested that it is feasible for industry to produce large quantities of deuterim by this cryogenic distillation method; it is, in fact, particularly well-suited for industrial areas where extensive supplies of hydrogen gas are available—near ammonia plants.

Flynn *et. al.* design of three small columns to operate at −423°F led to improved suggestions for a column or series of columns intended to increase the concentration of hydrogen deuteride in hydrogen from its natural abundance (one part in 3,500) to any desired amount. In designing their pilot plant to accomplish this specific job, C.E.I. scientists assumed that parameters obtained experimentally for an H_2-D_2 system would be applicable to a H_2-HD system. In order to obtain the necessary values, they varied the number and types of plates for the column. One column was packed with stainless steel helices and the other two fitted with perforated plates.

External Reflux Ratio

To adapt the column to a limited space, the designers chose a high external reflux ratio (20-40% greater than minimum calculated for their columns and derived from the vapour-liquid equilibrium curve calculated for a particular column). The result is a reduced column height and experimental value of the column is not downgraded.

Likewise, for space reasons, the experimenters made the number of plates as small as possible. Use of the McCabe-Thiele theory provided the engineers with the exact number of plates necessary.

The difference in column diameter between laboratory-scale and pilot plant-size equipment is somewhat arbitrary, it is reported. An increase from 1½ to 6 inches however places the column in the pilot-plant category. This pilot-plant size yields information directly applicable to commercial-size units. In terms of an equivalent heavy water production rate, the feed rate of this pilot-plant column represents approximately 45 lb. of heavy water for an 8,000-hour year.

The flow system of the column consists of three separate parts—circulation, condenser, and feed. In the circulation system, a heat pump, operating between the condenser and the reboiler, provides the necessary refrigeration at the top and associated boil-up in the reboiler. Hydrogen gas, the circulating refrigerant, transfers heat from the condenser to boiler. This operation supplies sufficient heat to fulfil the requirements of the system.

Condenser and reboiler systems are similar; the former has a finned heat exchanger and a reservoir, or heat sink, of liquid hydrogen. This furnishes the other portion of hydrogen needed for refluxing. As the reflux condenses, the hydrogen vaporises under atmospheric pressure. A resistance-type liquid-level sensing device and a solenoid valve on the transfer line keep the reservoir filled automatically.

Many of the problems encountered in designing this pilot plant were characteristic of standard distillation operations but a few arose which are peculiar to low-temperature units Thus, in high-temperature units, heat loss is from the column. As a result, some additional vapour will condense to a liquid within the column. This means an increase in the internal reflux ratio. In addition, this transfer of energy (away from the column) requires that the size of the reboiler be increased to compensate for this heat loss. In low-temperature operations the effect is just the opposite; that is, a decrease in the internal reflux and a corresponding decrease in the hydrogen-deuterium separation. Since stepping up the refrigeration efficiency of the system is expensive, C.E.L. engineers designed a compact "cold box", crowded with components, to minimise heat leak. The column and accessories are suspended inside a vacuum space, from a stainless steel tripod supported on a bottom plate. This practically eliminates two energy transfer problems: (1) When the vacuum is of the order of 10^{-6} to 10^{-7} mm. Hg, heat transfer by gaseous conduction is negligible. (2) Conduction through solids is small when supports are designed in the form of long, low-conductivity heat paths.

Radiation Heat Transfer

The problem of radiation heat transfer has been solved by using a set of insulating shields. Surrounding the column is an inner shield of thin copper sheeting. It is attached to the hydrogen reservoir, at the top of the column. Because of copper's high thermal conductivity, the design can provide a shield having the same temperature gradient as the column, top to bottom. Thus, heat radiated from the shield to the column is negligible. Reduction of the heat transferred to this copper shield from outside the column is effected by placing a liquid-nitrogen-cooled shield around the inner shield (2).

Proper allowance has also to be made for coefficients of expansion. For instance, some parts of the pilot plant, including the column, change in length by as much as one-half inch with the 280° K temperature change that takes place in the system. One method of relieving this situation is to solder short sections of bellows to one end of a line. Another way is to use a loop or a couple of bends, in a small diameter line, in order to place a short section of the line at right angles to the principal axis.

The only major item causing a change in design between this pilot plant and an industrial unit is the provision made for a supply of pure gaseous hydrogen to the column feed.

Bottom product and the distillate are joined together and circulated to the column as feed. Once this column has been charged, a closed circulation system is in force—no new feed is necessary. Other designs, however, would have to have an outside source supplying feed continuously to the column and purification apparatus for the hydrogen.

The designers also recommend traps of absorbents in liquid nitrogen baths or other purifying systems in the feed line to eliminate any plugging of the perforated and screen plates resulting from impurities in the hydrogen gas. Even small impurities can quickly reduce the column's separation efficiency they report. In fact, if there are sufficient impurities present to clog the plates, the distillation would cease.

Many safety features are incorporated in the C.E.L. design. An operating pressure of 1.3 atmospheres was selected. All electrical switches and motors are explosion-proof. All electronic, non-sealed equipment is constantly purged with nitrogen gas. All equipment is grounded. Besides placing the column in a well-ventilated room, the designers have made provisions for either piping the exhaust hydrogen to a gas holder outside the building or to release it through a vent in the roof. Other obvious safety precautions, such as no open flames, methods of reducing high-static potentials and use of non-sparking tools, are maintained. The successful operation of this unit suggests that many other problems in gas separation might be solved by adopting cryogenic techniques.

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Training in Dealing with Radiation Dangers

PLANS for courses to train graduates in science and medicine to undertake specialist posts in the field of radiological health and safety are announced by the U.K. Atomic Energy Authority.

These courses were recommended by a committee set up with Sir Douglas Veale as chairman after the Windscale accident of October 1957.

It is expected that two courses will be available at London University in October this year. Courses may also be held in Birmingham, Edinburgh and Salford.

Another part of the scheme to extend knowledge of dealing with radioactivity and give training in dealing with radiation hazards is a summer school to be held at Oxford in August. This is intended for people who, although not engaged in full time health and safety duties, need some knowledge of the problems involved.

Usefulness of Cold Probe Corrosion Test in Evaluating Fuel Treatments

PROBLEMS encountered in the burning of sulphur-bearing fuels in oil-fired boilers, and methods of alleviation were considered by Mr. H. N. Wigan, director, Combustion Chemicals Ltd., in a paper given at a meeting of hospital superintendent engineers.

Oxidation of SO_2 to SO_3 and the subsequent formation of sulphuric acid is a major cause of fouling and corrosion, stated Mr. Wigan. Factors influencing SO_3 formation and its level in flue gases were an increase of sulphur content of the fuel; an increase in excess air; an increase in flame temperature (up to approximately 1,750°C); an increase in furnace wall and generating tube temperature; and an increase in load. Hence the higher the efficiency, the harder the service and the heavier the load, the greater could be the problems created by the sulphur content of fuel. At the other extreme, however, intermittent operation with frequent shut-downs and operation at light and varying loads tended to make more trouble from corrosion and acidic build-up in the back-end.

To avoid low temperature corrosion, back-end temperatures could be re-used but this involved increasing excess air so that not only would more SO_3 form but combustion efficiency would be reduced. Also since SO_3 would not be allowed to condense out in the flue passages or inside the stack, more sulphurous gases would be emitted to atmosphere. A measure of success in alleviation of corrosion of the internal surfaces of metal stacks, and of acidic smut emission could be achieved by lagging the stack, for instance, by encasing it with an aluminium sheath. This, however, was costly. Chemical control over corrosion and fouling had been along three lines: neutralising SO_3 in flue gases; protection of metal surfaces against the effects of corrosive films; and inhibition of SO_3 at source, i.e. control of the oxidation of SO_2 to SO_3.

Neutralising SO_3

In neutralisation of SO_3, ammonia gas was injected into the flue gases. Results of trials in oil-fired plants in the U.K. had been erratic, but better success had been achieved on coal-fired plant. Chemicals such as magnesite (magnesium carbonate) and dolomite (magnesium carbonate and calcium carbonate) have been employed. These were injected by compressed air usually direct into the combustion zone.

To protect metal surfaces, coal tar bases have been injected into flue gases. Again results have been erratic, with aggravation of the accumulation of deposits resulting in additional fouling and failing to alleviate acidic smut emissions.

Latterly the Central Electricity Generating Board (C.E.G.B.) have turned more to the injection of chemical compounds in powder form comprising mixtures of magnesite and/or dolomite and certain metals, notably zinc. Zinc dust alone was being experimented with at certain C.E.G.B. stations, at ¼ lb. to 1 lb. per ton of fuel burned. This method, Wigan reported, had been successful in suppressing the acid dew-point, increasing availability and reducing acidic smut emission. Expensive metering apparatus was required, however, and the application of powders was restricted to only the largest boilers.

Fuel additives were as varied as their composition, Mr. Wigan remarked. They were usually added to the fuel oil in the proportion of 1/500 up to 1/4,000. They should eliminate sludge and prevent its further formation. Rendering sludge combustible was bound to show a saving in fuel. Apart from the desirability of keeping tanks and pipelines clean some additives reduced surface tension of the oil, thereby improving atomisation.

To prove the ability of Combustion Chemicals' fuel treatments to control SO_3 and alleviate sulphur corrosion and fouling, the company employed the Cold (or Water-cooled) Probe Test. (See also CHEMICAL AGE, 14 February, p. 281, for details of a radioactive corrosion probe.) This involved the use of a hollow mild steel thimble, 6 in. to 12 in. long, with a highly polished exterior surface attached at its open end to an assembly through which cold mains water passes. The thimble, weighed to four decimal places of a gramme, is inserted into the flue ducting, before the I.D. fan, through a hole drilled for the purpose. This thimble is left for a period of one hour, and on withdrawal is carefully washed and dried and then weighed. Loss of weight is usually found, representing the loss of metal due to corrosive attack during its insertion in the flue gas stream.

The same procedure was adopted after the fuel had been treated and the difference between the two losses in weight were compared, improvement on the second test was an indication of reduction in potential corrosion in the boiler system. While this was only a comparative test, Wigan stated that it had been established that there was a direct relationship between the reduction in loss of weight and the level of SO_3 in the flue gases.

Range of New High Purity Oxide Refractories Developed

AN exhibition of Purox oxide refractories was held at the Mayfairia Rooms, Bryanston Street, London W.1, recently. Recent development at Morgan Refractories Ltd., Neston, Wirral, Ches. —a subsidiary of the Morgan Crucible Co.—has resulted in laboratory ware in these highly refractory materials becoming available to a greater degree of purity and in a wider range of shapes and sizes than has been possible in the past.

The range includes alumina, thoria, magnesia and lime stabilised zirconia, and each of these is offered as being the highest quality available today.

Purox recrystallised alumina contains 99.7% Al_2O_3. The crystal structure is the result of heat work alone, there being no addition to induce or reduce crystal growth. The material can be supplied as tubes, sheaths, crucibles, bricks, tiles, die pressings and special shapes.

Analysis of the raw material used for the manufacture of Purox thoria ware is 99.9% ThO_2. This grade can be supplied in standard and impervious forms to the same analysis. Crucibles up to 250cc. can be supplied to order. Tubes and sheaths can also be produced, in rather restricted sizes at the moment.

Purox magnesia is available in the form of crucibles, tubes, bricks, tiles, and small die pressings. 100cc. crucibles are standard, and tubes and sheaths up to 800 mm. by 25 mm. by 20 mm. bore, and 458 mm. by 75 bore can be supplied to order. Analysis varies with the method of manufacture.

The zirconia is fully stabilised and contains 97% ZrO_2. Crucibles up to 500 cc. are standard, and tubes and sheaths up to 800 mm. by 25 mm. by 20 mm. and 525 mm. by 8 mm. by 5 mm. can be supplied to order.

Purox sintered pure alumina is made from the same material as recrystallised alumina, but it is fired to a lower temperature and is, therefore, not impervious.

Ware Purox often required to very accurate dimensions, and grinding facilities are available to meet such requests. Plus or minus 0.001 in. can be achieved readily, and both outside diameter up to 8 in., up to 3 ft. long, and bores of up to 6 in., up to 1 ft. long, can be ground.

Shell Group Spend £10 m. on Chemicals in U.K.

PURCHASES made in the U.K. last year by the Royal Dutch/Shell group of companies included £10 million on bulk chemicals and catalysts for refinery use. Radioisotopes were bought for use in research.

Certain purchases for the chemical side of the business are not included in this figure nor in the £39 million total orders placed with British industry.

Purchases for U.K. refineries and research establishments increased from £12 million in 1957 to £12.5 million last year.

O.C.C.A. POLYMER CONFERENCE—3

Toxicity of Epoxides and Diisocyanates Discussed by Dr. M. H. C. Williams

HAZARDS associated with indiscriminate use of certain members of both epoxides and diisocyanates were considered by Dr. M. H. C. Williams, Imperial Chemical Industries Ltd. Of the epoxides, Dr. Williams pointed out that it was the same cross-linking property of the epoxides which was attractive to plastics technologists that had brought them to the attention of cancer research workers. Many short chain bisepoxides possessed the property of interfering with cell division (*mitosis*).

Polymers of the diglycidyl ether of diphenylolpropane have been found to be non-toxic to bone marrow and not carcinogenic. Diglycidyl ether of diphenylolpropane itself has proved to be totally inactive as a carcinogen in mice and to have no effect on bone marrow. So far, too, all monoepoxides often used as diluents in industry which have been tested as tumour inhibitors, have proved inactive. Dermatitis from epoxy resins could be combated by good personal hygiene.

Aromatic Amines

Certain aromatic amines such as metaphenylene diamine and diamino diphenyl sulphone had been introduced recently as hardeners. Technologists, suggested Dr. Williams, might unwittingly experiment with carcinogenic aromatic amines which were already known or strongly suspected to cause bladder tumours in mice. He had recently heard benzidine, a proven human carcinogen suggested as a good hardening agent. He would regard the use of carcinogenic aromatic amines as hardener in epoxy systems as highly dangerous. Anyone engaged in a search for new aromatic hardeners was recommended to consult the paper by Walpole and Williams (*Brit. Med. Bull.*, 1958, **14**, No. 2, 141).

It had been demonstrated that the cytotoxic and tumour inhibitory action of vinyl cyclo hexene dioxide gave rise to skin tumours after painting an industrial sample of this chemical on mice. Diphenylolpropane diglycidyl ether had no skin carcinogenic action for mice. Four skin tumours had been produced in 30C3H mice, however, afer painting a 5% solution of an aliphatic diglycidyl ether. A similar bisepoxide, gycerol diglycidyl ether has been found to be toxic to the bone marrow of monkeys and rats.

Resorcinol diglycidyl ether was a tumour inhibitor and also was toxic to the bone marrow of monkeys and rats. Pentaerythritol diglycidyl ether possessed similar toxic properties as did pentaerythritol tetraglycidyl ether. Diglycidyl adipate had only a feeble toxic action on bone marrow and diglycidyl sebacate no such action. Skin painting tests on mice were now in progress. Phenyl glycidyl ether and other monoepoxides had not yet been extensively tested for carcinogenic activity, but they were inactive as inhibitors.

It was pointed out by Dr. Williams that it was most difficult to extrapolate from these animal experiments to man. Blood counts of industrial and laboratory workers exposed to bisepoxides having a toxic action on bone marrow had revealed no case of reduced blood components. In modern conditions of manufacture and use, he stated, there was no indication of gross absorption of these chemicals. They had a low vapour pressure and unless high temperatures were created a vapour hazard from such bisepoxides was unlikely. Ventilation of ovens outside the working space, however, was wise.

With regard to the possibility of skin tumours arising in exposed workers, it was a wise precaution to institute routine skin examinations. In Britain it was fortunate that the general sale of the toxic and carcinogenic bisepoxides had been delayed, while further toxicological evidence was collected.

Diisocyanates were not believed to possess any carcinogenic properties, although they varied considerably among themselves in toxic properties. Certain members of the series produced allergic states and respiratory tract irritation and if recommended precautions were not taken in some processes, the toxicity of certain volatile diisocyanates could handicap their industrial application and development.

T.D.1 or 2:4-toluylene diisocyanate gave rise in trivial atmospheric concentrations to tightness of the chest, soreness of the throat and coughing at night in some exposed persons. Reaction products in T.D.1 and certain polyalcohols had been developed, but as these products contained unreacted T.D.1, this was sufficient to cause trouble particularly in spraying under poorly ventilated conditions.

A maximum allowable concentration of 0.1 p.p.m. for toluylene diisocyanate had been suggested (Zupp, J. A., *A.M.A. Arch. Indust. Health*, 1957, **15**, 324) and Dr. Williams agreed with this figure for the exposure of new personnel. For persons previously sensitised by exposure or with an asthmatic history, lower levels gave rise to bronchiospasm. Contaminated boots and vessels should be decontaminated, and spillages quickly attended to. Suitable decontaminant liquids were:

Liquid Decontaminant	*Parts by Volume*
Industrial methylated spirits	55
Water	40
Concentrated ammonia S.G. 0.88	5

Solid Decontaminant	*Parts by weight*
Sawdust	25
Kieselguhr tech.	37
Industrial methylated spirits	20
Triethanolamine	4
Concentrated ammonia S.G. 0.88	4
Water	10

Diluted ammonia	*Parts by volume*
Concentrated ammonia S.G. 0.88	1
Water	10

Emphasising decontamination of tools, or spray guns, Dr. Williams recorded the case of one man who developed tightness of the chest having put a screwriver contaminated with T.D.1 in the top of his overalls.

No vapour hazard had been encountered even under the most adverse conditions when rigid foam based on 4:4′-diisocyanotodiphenyl methane was produced *in situ*.

Dermatitis due to diisocyanates was considered extremely rare. Cases noted had been in persons who had removed reacting resin with acetone. Solvents such as acetone, etc., appeared to carry the isocyanates through the skin. Dr. Williams recommended cellosolve for this purpose.

Previous reports of the O.C.C.A. Biennial Conference held at Edinburgh appeared in CHEMICAL AGE, *9 May, p. 781, and 16 May, p. 827.*

Aspects of Polyurethane Chemistry Discussed by I.C.I. Dyestuffs Research Teams

DEALING with some aspects of polyurethane chemistry, Dr. R. J. W. Reynolds and Dr. H. Gudgeon, research department, Dyestuffs Division, Imperial Chemical Industries Ltd., considered that with the extended interest shown in polymers there had developed also a deep appreciation of the dependence of the properties of these materials on the chemical and macromolecular structures.

Among the newer polymers were the polyurethanes, formed by reaction of various intermediates with isocyanates and a class outstanding in providing a wide range of properties, the properties being dependent primarily on the nature of the intermediates employed, the degree of branching or cross-linking in the molecules, and sometimes, even in actual method of fabrication.

Fibres. No polyurethane had attained any large-scale importance as a fibre-forming polymer, although one based on 1:4-butanediol and hexamethylene diisocyanate (Perlon U. m.p. 184°C) was made for bristle, monofil applications and for moulding.

Elastomers. Formation of a resilient rubber by compounding Vulcaprene A on aldehyde liberating substance such as hexamethoxy-methylamine was noted by Reynolds and Gudgeon. The cured rubbers obtained had only moderate mechanical

properties but excellent resistance to oils and petrol and to oxidative ageing. Curing of Vulcaprene could also be effected by treatment with a diisocyanate. Compositions cured in this way were used as flexible lacquers with good resistance to flexing and abrasion and as cold-curing adhesives.

Recently considerable efforts had been made to modify this type of process so as to obtain products that could be processed by conventional methods and had the excellent mechanical properties typified by those of Vulkollan. In some of the products developed the main component was a crystallisable, linear polyester (e.g. polyethylene adipate) in others crystallisable polyethers (e.g. polytetrahydrofuran) with hydroxyl end-groups, tolylene diisocyanate being used for chain extension.

Flexible and rigid foams. Another class of products which was at present of great commercial prominence were the flexible foams, prepared from adipic acid, diethylene glycol and a small proportion of trimethylolpropane and as the diisocyanate, mixtures of tolylene diisocyanate isomers were preferred.

During the past year or two the use of polyethers with hydroxy end-groups had assumed major importance, stated these I.C.I. workers, in particular the polyethers which could be obtained by the alkali-catalysed polymerisation of polypropylene oxide.

Elastic Polyurethane Foams

Elastic polyurethane foams with excellent mechanical properties had also been prepared from the hydroxyl-ended linear polyether obtainable by polymerisation of tetrahydrofuran castor oil and polymerised unsaturated natural fatty acids had also been used as intermediates for the manufacture of polyesters for polyurethane foams.

The more highly branched the polyesters or polyethers used in making rigid foams, the less flexible and resilient were the foams obtained. Such rigid foams were likely to find substantial outlets as insulating and structural reinforcing materials. Because the foams had to be generated *in situ* in small spaces and cavities, where provision of good ventilation was difficult, the use of diphenylmethane diisocyanates presenting a low toxicity hazard were favoured in the U.K.

Surface coatings. The simple procedure used in applying coating compositions based on a branched polyester and a polyisocyanate had a number of disadvantages: the short pot-life of compositions; exclusion of moisture and hydroxylic solvents, and possibly poisoning by volatile isocyanates. The toxic hazard could be reduced by using less volatile polyisocyanates, or by reducing the effective vapour pressure of tolylene diisocyanates, (e.g. by combining three molecules with one of hexanetriol or trimethylolpropane).

Chemical reactivity and processing. Although a great deal of effort had already gone into the elucidation of how physical and chemical factors influence the building-up of polyurethanes, much remained to be learned and it was still only possible to deal with them in rather empirical terms.

It was possible to moderate reactivity of polyesters to some extent and thus delay gelation on reaction with a diisocyanate by using primary-secondary glycols such as 1:2-propylene glycol or 1.3-butylene glycol in their formulation instead of ethylene glycol or diethylene glycol, resulting in the polyester containing a proportion of the less reactive secondary hydroxyl groups.

Reactivity of isocynates might be varied over a very wide range by choice of nucleus, but economies limited the number available for commerce, the most important being mixtures of the isomeric 2.4-2:6-tolylene diisocyanates and diphenylmethane diisocyanates.

Isocyanate chain reactions are kinetically complicated: the reaction with alcohols, for example, is autocatalytic and very sensitive to the nature of the medium in which it proceeds. The reaction with water was particularly important: it resulted in the tying together of isocyanate ended chains through urea links and these urea groups themselves could undergo further reaction leading to branching and ultimate cross-linking. Under some conditions the isocyanate end groups could trimerise again introducing branching and cross-linking: this reaction was usually considered to require a basic catalyst, but it had been found to occur also in the absence of bases.

Reaction between isocyanates and hydroxyl groups could be catalysed by a diversity of agents. For some purposes it was desirable to use volatile tertiary amines, which could evaporate from the system soon after fulfilling their function. In other applications there appeared to be some advantage in using tertiary amines containing groups reactive towards isocyanates so that they become permanently built into the polymer structure. It was now clear that reaction of isocyanates with hydroxyl groups could be favoured relative to reaction with water by using certain basic catalysts (e.g. triethylene diamine) or certain metallic compounds (e.g. dibutyl tin dilaurate).

Retarding the Reaction

Retardation of reaction of isocyanates to provide more latitude in operation was not as easy to achieve as catalysis. Some acid chlorides had been used to extend the pot-lines of some lacquer compositions and small traces of some stronger acids, e.g. phosphoric acid, would retard reaction but since larger amounts functioned as catalysts, the balance was delicate for practical use. There was evidence that acidic retarders, even in small amounts in lightly cross-linked structures, such as rubbers, might accelerate hydrolytic degradation. Nearly all these factors were of critical importnace in the manufacture of cellular polyurethanes.

Effect of structure on properties. Linking together of linear or essentially linear low-melting aliphatic polymers of fairly high molecular weight afforded elastomers, whereas polymers with numerous branches carrying isocyanate-reactive end-groups and capable of giving a close network structure afforded stiff materials. Urethane rubbers with high tensile and tear strength and resistance to abrasion were characterised by a streamline backbone structure, the units of which could crystallise readily when not inhibited in some way.

Crystallisation on stretching was common in some elastomers, including natural rubber, but in those based on polyethylene adipate crystallisation set in at much smaller elongation (50%) and occurred to a greater extent than in other rubbers.

Polyurethane coating compositions prepared from branched polyesters and polyisocyanates were also remarkably tough. In their case the versatile reactivity of polyisocyanate might be playing an important part by reacting also with reactive groups in the coated substrate, thus supplementing good film properties by the excellent adhesion characteristic of the polyurethane coatings. The lower limit of the temperature at which rubbers and films remained flexible was also determined largely by the nature of the backbone structure, i.e. the glass/rubber transition point of the parent polymer.

Slow Crystallisation

For some rubbers which crystallised readily on stretching, a temperature of 0°C might suffice to induce slow crystallisation on storage in the unstretched state. leading to hardening. More commonly, however, it was not the transition from rubbery to crystalline, but that from rubber to glassy state which limited the temperature at which films remained flexible. The greater the proportion of aliphatic hydrocarbon segments in the chains (provided the structure did not lend itself to crystallation) the lower the temperature at which the polymer stiffens. Incorporation of natural fatty acid residues, e.g. ricinoleic acid, was a convenient method of improving low temperature flexibility.

In making rigid foams, incompatibility of ingredients, or premature setting up might set a limit to the extent of branching of the resin that could be tolerated. Attempts had been made to overcome this by introducing a secondary cross-linking mechanism which only begins to operate at the high temperatures attained at the end of the foam-making step.

Some of the isocyanate-derived links were thermally unstable at about 150°C. The polyurethane link from an aliphatic hydroxyl group was sufficiently stable at 150°C but biuret cross-links formed by addition of an isocyanate to a chain urea link dissociated more rapidly. Isocyanate links formed by trimerisation of isocyanate ends would be quite stable under those conditions.

Permeability of polyurethanes to moisture was important. In rubbers and flexible foams it was necessary for maximum resistance to hydrolytic breakdowns to minimise concentration of water in the polymer. Higher proportions of amide groups in polyesteramide intermediates favoured water absorption; fatty acid ingredients probably conferred the greatest stability by virtue of their hydrophobic nature.

ANALYSIS OF TAR ACIDS—2

Spectroscopic and Chromatographic Methods Described by Willcock

SPECTROSCOPIC methods were considered next. All phenols had similar U.V. spectra since most of their absorption was due to resonance of the benzene ring. Some degree of correlation with substitution pattern was possible. Because of this low order of characterisation of phenols by U.V. measurement and the fact that specific extinction coefficients of phenols were approximately the same, total phenols contents of solutions were readily and accurately determined by measurement of absorption at 2,800 Å. This assumed, of course, that the solvent had no strong absorption at the same wavelength. Such measurements might be made in aqueous solution, which permitted determination of phenols content of a hydrocarbon oil by U.V. analysis of caustic soda extract of the oil. In alkaline solution, a shift and intensification of the phenol U.V. spectrum occurred and measurements were made at appropriately higher wavelength (2,900 Å) for highest sensitivity. The method had wide application when total phenols content was required and, for this purpose, it was probably the most rapid and reliable method.

I.R. Spectroscopy

I.R. spectroscopy was extremely valuable for identification of phenol compounds. More important, it might be used for quantitative analysis by measurement of absorption intensity at a frequency characteristic of the substance being determined and relating these measurements to a previous calibration using solutions of the pure component of known strength. A mixture of phenols could be analysed by this method, providing a characteristic absorption frequency could be found for each component and absorption at that frequency varied linearly with concentration. Obviously, the more complex the mixture, the less chance of finding a suitable wavelength, but six component mixtures of cresols and xylenols had been satisfactorily analysed.

Of the methods mentioned so far, I.R. spectroscopy was the only one capable of dealing with mixtures of phenols, although any of the others might be applied to single phenols in solution. It was apparent, therefore, that if a technique was available for separation of phenolic mixtures, the separated components might be readily determined. Chromatographic separations of various kinds were, therefore, receiving increasing attention. Such methods could be very elegant, requiring a very small amount of material for analysis and not necessarily involving expensive or complicated apparatus.

Bulenburg first applied adsorption chromatography to phenols analysis by chromatographing the azo-dyes on alumina but this had the disadvantage that each phenol could give more than one dye. Fractional distillation in conjunction with chromatography (**20**) had been used to obtain a partial analysis of xylenol mixtures, while Cooper and Commins based their method on separation of phenol ethers.

Paper chromatographic methods (**21**) had been widely and successfully used for separation and identification of a wide variety of phenolic mixtures. It was difficult to isolate the phenols from the chromatogram so that they might be determined, and losses from evaporation could occur. Such methods were, therefore, of very limited value for quantitative work.

Liquid-liquid chromatography was first applied for determination of phenol in cresylic acid (**22**), using water on silica gel as fixed phase and cyclohexane as mobile phase. Celite (**23**) was a more suitable support, because silica gel adsorbed phenols, and better separation could be obtained using a buffer salt solution, such as sodium silicate or sodium phosphate, instead of water.

Pearson separated phenol, cresols and some xylenols by means of aqueous methyl cellosolve on silica gel, while water on silica gel and sodium silicate on Celite had been used for separations of di- and monohydric phenols respectively (**24**). Mr. Willcock and his colleagues had worked along similar lines to the two latter approaches, and encouraging results had been obtained.

To simplify identification and analysis of phenols in eluates from chromatographic columns, the eluate had been passed through a small cell and its U.V. spectrum at 2,800 Å measured and recorded continuously. A modification to the Unicam S.P. 500 instrument (**25**) had enabled the latter to be used very successfully

Emergence of a phenol from the column was indicated by the appearance of a peak on the recorder chart. The area under such a peak was proportional to the concentration of phenol present. Integration of areas might, therefore, be used to give an analysis of the mixture or, alternatively, the eluate might be collected in fractions, appropriate blends made as indicated by the chromatogram and then phenol contents determined by U.V. absorption. A solution containing about 1 mg. of each phenolic component was required for this type of analysis. The phenols might be in organic or aqueous solution, but in the latter case the sample should be slurried with twice its weight of Celite, which was then added to the top of the column.

In the case of dihydric phenols, catechol, resorcinol and quinol were readily resolved by silica gel-water and their homologues gave either separate peaks or peaks containing simple mixtures which might be collected and examined. At the present time, monohydric phenols up to and including the xylenol range might be resolved into phenol, each of the cresols and 2 : 5- and 2 : 6-xylenols, while other xylenols and ethyl phenols occurring naturally with them were only partially separated, usually as binary mixtures. Future work would be directed towards extension and improvement of this resolution by liquid-liquid chromatography, possibly in conjunction with other techniques.

Gas-liquid chromatography (G.L.C.) had been used extensively in Mr. Willcock's laboratories and offered a rapid and simple technique for solution of many analytical problems, although there were many limitations still to be overcome. Accepting these limitations, much use might be made of G.L.C. for analysis of tar acid fractions, particularly low boiling ones. For example, phenol, *o*-cresol, 2 : 6-xylenol, *m*- plus *p*-cresols and 2 : 4- plus 2 : 5-xylenols might be very rapidly determined in their mixtures by this technique.

G. and G. Apparatus

Mr. Willcock normally used a silicone oil on Celite as fixed phase, with column temperatures between 140° and 200°C., depending on boiling range of the phenols. The Griffin and George Mark II G.L.C. apparatus, employing katharometer detection, had been found very satisfactory for the purpose. In quantitative analysis, area integration of the peaks was insufficient and an internal standard was normally used to obtain accurate results, since katharometer response decreased with increase in boiling point of the phenol. Accuracies to within ±1% of the true value were obtainable.

If no improved fixed phase was found, it might be possible to deal with partially resolved mixtures by condensing them from the gas stream from a large G.L.C. column and analysing by I.R. spectroscopy. Alternatively, with the advent of extremely high efficiency columns, such as had been used for hydrocarbon separations (100-150,000 theoretical plates), it was probable that even close boiling isomers, such as *m*- and *p*-cresols, would be separated, irrespective of the fixed phase.

In conclusion Mr. Willcock said that, while many methods were available for determination of tar acids, the majority were non-specific. Some simplification of tar acid mixtures was, therefore, necessary before application of such methods. Chromatographic techniques appeared to offer the best means of achieving this.

First part of our Report of the Paper given at a recent S.A.C. Midlands Section meeting by Mr. H. G. Willcock, appeared last week, p. 831

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Triethylenediamine Provides Balanced Catalysis for 'One-shot' Polyether Foams

WORK on flexible 'one-shot' polyethers with respect to machine formulations data has recently been extended by Houdry Process Corporation, Philadelphia, U.S. In particular, detailed information has been released on the use of Houdry's urethane foam catalyst, Dabco (triethylenediamine). It is claimed that when triethylenediamine is used as the sole catalyst in 'one-shot' polyether systems thermal and hydrolyte stability of the resulting foam is outstanding. Also the cost of raw materials for production of such flexible foams using the indicated formulation is approximately $0.40 per lb. of foam.

Triethylenediamine is reported as having a marked activity for catalysing reactions of isocyanates with active hydrogen compounds. Thus, a model system combining phenyl isocyanate and 2-ethylhexanol can be accelerated up to 10 times more effectively by this catalyst. Of theoretical significance is the fact that this catalyst is not a strong base, although it is found to be more active than appreciably stronger bases. This high activity is believed to be due to steric factors, such as its compact structure which make the electron pairs located at bridgehead nitrogens readily accessible. The catalyst's high degree of symmetry provides also for additional effect in chemical reactions.

In the 'one-shot' polyether system triethylenediamine is found to provide the balanced catalysis—chain extension, cross-linkage and gas evolution which proceed at optimum rates relative to each other—which is vital to producing a successful foam.

Formulation

A formulation has been worked out to produce open-moulded flexible 'on-shot' polyether foams on a Hennecke-type foam machine. This machine uses high pressure Bosch pumps to inject toluene diisocyanate and actuator streams, and is designed primarily for mixing very high viscosity prepolymers. The foam formulation which is run at a total flow rate of 12 lb./minute is as follows:

Component	Amount
Polypropylene glycol 2,000	72.0
*Max. triol LG 56 or Puracol TP 2,540	28.0
Toluene diisocyanate (80-20)	38.0
Water	2.9
Triethylenediamine	0.5
Silicone X-520	0.5

* The polyol component can be varied up to 100% triol depending upon desired physical properties.

The actuator is prepared by dissolving triethylenediamine in water and then mixing in the X-520 stabiliser. The toluene diisocyanate (TD1), polyether and actuator mixtures are pumped to the mixing chamber as four separate feed streams. A 'ribbon'-mixer consisting of 3/16 in. flat steel stock, twisted to rotate 180° in a 4-in. length is substituted in place of the conventional multipin rotor. This 'ribbon-mixer' is welded to the shaft and machined to close tolerance of the mix chamber wall, the purpose being to reduce mixing and minimise shear. Mix speed used is varied in the range of 650 up to 2,000 r.p.m. In addition, substitution of low pressure springs on the TD1 injection nozzles (opening pressure of 60 lb. or less) is recommended. Overmixing, Houdry report, results in foams which are slow to develop strength or in complete collapse. It has also proved advantageous to introduce all the catalyst with about a third of the water into the upper portion of the moving chamber. The remaining water and silicone X-520 are pumped into the lower portion of the mix chamber. This has been found to delay gas evolution and to give the chain extension and cross-linking portions of the reaction a head start.

Extension nozzles used have varied from 6-20 mm. in diameter and were either 18 mm. or 120 mm. in length. Increasing the nozzle length and/or decreasing the nozzle diameter results in increased cell size in a given foam. Foaming is carried out into a 12 in. by 12 in. by 14 in. corrugated cardboard box. It has proved possible to 'free-blow' as high a block as 30 in. without signs of internal scorching, it is reported.

Odourless Foam

The foam produced is odourless during and immediately after production (cream time, 8-10 seconds and rise time, 60-80 seconds). The cells formed are described as fine and uniform in appearance and virtually 100% open. Crushing, ordinarily not needed to prevent shrinkage, may be desirable to improve resiliency and other physical properties. Under conditions of high relative humidity foams are found to be tack free in about 15 minutes at room temperature. Increasing temperature and humidity can drastically reduce tack free time. It is found that suitable cure may occur after 24-48 hours at room temperature.

Due to the flow characteristics of the very low viscosity 'one-shot' polyether systems, it is considered that these should be very useful not only in slab stock production, but in close moulding of intricately-shaped foams. The low-viscosity differential between components should also aid in solving some of the problems associated with spray foaming, both flexible and rigids, and in similar applications, the high activity of triethylenediamine should prove advantageous.

Typical Physical Properties of Foam

*Density	2.0—2.3 lb./ft.³
Compression set at 50% deflection (22 hours)	6%—8%
Compression — deflection at 25% deflection	0.35—0.40 lb/in.²
Tensile strength	16.0—20.0 lb./in.²
Tear strength	3.5—4.5 lb./in.

* Lower densities can be obtained, it is stated, by increasing amounts of water and TDI used, by slight increases in triethylenediamine concentration.

Approximately 0.3 parts of triethylenediamine per 100 parts polyether is an average concentration value. Where higher percentages of triol are used, less catalyst is required. For best foam properties and ageing characteristics, use of the catalyst is recommended in conjunction with X-520 silicone as foam stabiliser. Foams can be produced using slightly less triethylenediamine (0.2-0.3) and 0.05-01 parts of organotin catalyst plus the usual silicone X-520.

One-shot polyethers can also be made, Houdry claim, with low pressure foam machines utilising either two or three feed streams. In two-component equipment actuator solution is premixed with the polyether and then brought into contact with TDI in the mixing chamber. If the machine is equipped with three feed streams, the activator is pumped as the third feed stream and is mixed with TDI and polyether in the mixing head.

Foams Blown by CO_2

'One-shot' polyether rigid foams have been produced both on a laboratory hand-mix scale and in a foam machine such as the Hennecke-type. In both cases, the foams were conventionally blown by CO_2 evolved from the reaction of water and excess isocyanate.

Component	Amount (parts by weight)
G2410 (Hydroxyl 495)	100.0
TDI (80—20)	82.0
Water	2.9
Triethylenediamine	0.4
Silicone X-520	0.5

Activator component (triethanolamine, water and X-520) is premixed briefly and added to the polyol-TD1, stirred at 900 r.p.m. for five seconds and poured into a suitable mould. The foam is stated to be tack-free as soon as rise is completed (about 1 minute) and no shrinkage occurs. Density is in 1.5-2.0 lb./ft.³ range.

Some limited investigative work has been done on polyether prepolymers by Houdry but foams with really low compression set values have not been successfully produced. However, prepolymer foam made with triethylenediamine and polyether prepolymers produced by Isocyanate Products Inc. are reported to be outstanding in all respects including compression set values.

A great deal of work on dimer acid ester foams has been carried out at several U.S. companies including Witco Chemical Co., Pittsburg Plate Glass and Naugatuck Chemical Division, U.S. Rubber Co. Good results in a density range of 1.4 to 2.2 lb./ft.³ are claimed. It also appears to be possible to prepare copolymers of dimer acid esters with polyethers using the 'one-shot' technique by pre-blending the polyols. The same is said to be true of adipic acid esters and polyethers.

Agents for Dabco triethylenediamine in the U.K. are Jacobson Van Den Berg and Co. (U.K.) Ltd., 3-5 Crutched Friars, London E.C.3.

Overseas News

FORMATION OF INTERNATIONAL PHARMACEUTICAL GROUP IN C.M.

UNDER the name Groupment International des Industries Pharmaceutiques,—G.I.I.P., national organisations representing the manufacturers of medicinal drugs in the Common Market countries have founded an international society.

In the Treaty of Rome, Article 195 refers to the possibility of Government consultation of representative European organisations in the various economic and social sectors. As there is a possibility that harmonisation of the public health laws in the various countries may be embarked upon in due course, the G.I.I.P. offers itself as such a representative organisation in the pharmaceutical field.

Headquarters of G.I.I.P. have been provisionally established in Brussels, and it has already contact with the appropriate officials of the E.E.C. organisation.

Rhineland Refinery Begins Production

The first of the new oil refineries of the Rhine-Ruhr area, that of Esso A. near Cologne, has begun production. The refinery, which has a throughput of 3,500,000 tonnes of crude oil annually and cost DM.235 million (about £19½ million), will supply ethylene to its main customers through a 56-mile pipeline. These customers are Chemische Fabrik Holten G.m.b.H., Oberhausen, Ruhrchemie A.G., of Oberhausen, and Chemische Werke Hüls, A., of Marl. The Munich chemical producers Wacker Chemie G.m.b.H., are to build a branch plant on the Cologne site for the processing of petrochemical by-products.

Hungarian Nylon-6 Plant in Production

An experimental plant for the production of nylon-6, the first in Hungary, which was erected with East German help, has now been handed over to the Hungarian authorities. Daily production of the plant, expected to be the forerunner of one or more commercially producing installations, is 1 tonne; this is soon to be raised to 2 tonnes.

Australia's Policy on Sulphuric Acid Production

Recommendations by the Tariff Board of Australia designed to increase sulphuric acid production by Australian industries from local materials, including pyrites and lead sinter gas, have been rejected by the Government.

In the report given to the Australian Parliament, Mr. McEwen said that the world sulphur position had become easier and that there had been substantial reductions in overseas prices. The Government had therefore concluded that, in view of the extra costs involved in using pyrites and other Australian materials, economic reasons no longer justified the continuation of past policy.

Pending the result of a new inquiry by the Tariff Board ordered by the Government, the present Bounty Act and regulations would continue for a year from 1 July. At its new inquiry the Board would be asked to recommend rates of bounty which, without encouraging the further conversion of Australian plants, would honour the Government's obligations to those in the industry who had co-operated in the past.

West Germans to Build Soviet Plant

A West German consortium led by the Essen firm of Ferrostahl AG has been given a contract worth DM.10 million (about £834,000) for the equipping of a chemical plant to be built in the Soviet Union. Other firms in the consortium are Benno Schilde AG, of Bad Hersfeld, and Voith, of Heidenheim. Apart from this order, the Soviet Union is at present placing a series of contracts with foreign firms for synthetic rubber plant.

Rumanian Plants Start Production

The sulphuric acid and fertiliser works at Navodari, in Rumania, have started operations at full capacity. The plant was built to Soviet designs and contains much equipment from the U.S.S.R.

At the Savinesti plant the first synthetic fibre unit has gone into production.

More U.S. Polythene Expansion Plans

Because of the growing market for polythene plastics pipe, Allied Chemical, U.S., are planning a 50% increase in capacity for high pressure polythene at Tonawanda, New York, and a new low pressure process plant at one of six possible sites in the Mississippi-Louisiana-Texas area. As they are now using all available ethylene in Tonawanda, Allied Chemical are negotiating to import 10 million lb. per year from a major Canadian refinery.

Texas Eastman have also announced that they are increasing polythene capacity at their Longview, Texas, plant from 85 million to 100 million lb. per year. These plant additions are expected to be ready before June.

Continuous Silicon Polymerisation

According to the General Electric Co. of the U.S., continuous silicon polymerisation has been made possible by the introduction of its new catalyst tetra-n-butylphosphoniumhydroxide. Disintegration of this compound takes place at a rather higher temperature than that under which polymerisation takes place. This means that a reaction mixture of low-molecular siloxanes can be pumped after polymerisation from one container into another, in which the catalyst—which constitutes only a few hundredths of one per cent of the mixture—can be destroyed thermically. The disintegration products resulting are inert and do not need isolating.

Start-up of Nitric Acid Plant in Israel

The Engineering Division of Société Belge de L'Azote et des Produits Chimiques du Marly (S.B.A.), Liège, has carried out successfully the start-up of nitric acid production plant erected in Haifa for Fertilizers and Chemicals.

Soviet Fertiliser Plants to Use Natural Gas

Two plants for the production of nitrogen fertilisers from natural gas are to be built in the Ulbek Republic of the Soviet Union. The first section of one of the plants—at Fergana, near a large oil refinery—is to start production in three years. The other plant, at Navoy, will produce, in addition to fertilisers, raw materials for artificial wool fibre. It will start working in 1963.

Belgium to Build French Styrene Plant Extension

The Engineering Division of Société Belge de L'Azote et des Produits Chimiques du Marly (S.B.A.), Liège, has been given a contract for the extension of the styrene plant of the Ethyl Synthese Co. The plant at Mazingarbe, North France, was built by S.B.A. with Koppers Inc., U.S.

U.S. Building Carbon Black Factory in Holland

The carbon black factory, which the U.S. Continental Carbon Company is to build near Rotterdam in Holland will have an initial capacity of 15,000 tons a year. Building costs are estimated at just under £1 million.

Already under construction in the same area is a carbon black factory which belongs to Ketjen Carbon N.V., a subsidiary of one of Holland's leading chemical concerns. An increase in the planned production capacity of this factory was recently announced—from 13,500 tons to 27,000 tons per annum.

Consumption of carbon black in Holland has fluctuated around 5,500 tons a year, with a peak of 6,000 tons in 1957, for the last few years. About 90% of this is used by the rubber industry, notably in the manufacture of tyres.

By far the largest part of the future carbon black production in Holland will have to be exported. It is expected that these exports will not only go to the other countries of the European Common Market. The Continental Carbon Company will be part-owners for 50% in another carbon black factory to be built near Bordeaux in the course of this year. Construction of U.S. Continental Carbon's plant will start immediately, and should be completed early in 1960.

NEW DYE FOR POLYESTER FIBRES SHOWN AT HANOVER

POLYESTREN, a dye for polyester fibres and polyester-cellulose mixtures was shown by Cassella Farbwerke Mainkur AG, Frankfort on Main, at the recent Hanover Fair.

Heating is by a thermosol process, taking between 40 and 50 at a temperature of 210-215°C.

Polyestrendruck dyes have also been introduced for printing on polyester and polyester-cellulose weaves. Other new materials shown by Cassella included a new type of the melamine-based resin Maprenal, which permits the manufacture at low temperatures of burning-in lacquers such as those used by the vehicle and toy industries, and a variation of the melamine-based resin Medurit which permits the production of colourless and virtually non-inflammable film with high resistance to chemicals and good electrical insulation for a range of melamine mouldings. This is marketed under the name of Madurit T2 extra powder.

Two new pigment dyes for the colouring of plastics and lacquers were Indanthrenbrillantbordo (Bordeaux red) RRL konz. Plv. and Indanthrenscharlach (scarlet) GL Plv.

CIBA AG, Basle, exhibited a wide range of Araldit resins, filling media, hardeners, wax foils, etc., for the manufacture of extra-resilient plastics for machine tools and industrial models, the Süddeutsche Kalkstickstoff-Werke AG had an attractively-displayed show of the base materials of which it is one of Europe's leading producers — dicyandiamide ($C_2H_4N_4$), benzoguanamine ($C_9H_9N_5$) and melamine itself ($C_3H_6N_6$)—and the E. Merck AG concern of Darmstadt showed thioglycol acid esters for the plastics industry.

Cellulose Foil

Wolff and Co., of Walsrode, displayed a cellulose foil, Transparit, into which bread can be placed for baking, the bread being taken from the oven ready packed for sale. The oldest nitro-cellulose plant in Federal Germany, Wolff also produces collodium wool, new types of methyl cellulose (Walsroder MC), special foils one side of which is coated with a polythene layer and one foil composed of one layer of wax situated between two of polymer-coated cellulose. Special silicon-based filtration media for the cellulose industry and a newly-developed anti-foam agent, said to be the first in the world, for the sulphate-cellulose industry, were shown by the Hamburg firm Schill and Seilacher.

Carl Huth and Söhne, of Bietigheim, had on show a wide range of washers, membranes and other spare parts for pumps and other plant made from the Du Pont plastic "Teflon"; these are proof against concentrated sulphuric and other acids, caustic soda, benzene, oxygen, chlorine and many other substances and gases as well as being able to bear temperatures of between −200°C and +260°C. Rehau Plastiks GmbH, producers of piping for the chemical and other industries from p.v.c. and other plastics, have developed a new washer fitment for use on the internal joins between male and female plastic pipe ends, the fitment insuring extra fastness in the concern's waste liquids and other pipes.

A wide range of new synthetic resin-based lacquers was displayed by the Wuppertal firm of Dr. Kurt Herberts and Co. These included Europe's first-ever dispersible wire enamel on an acrylonitrile-polymerisation resin base, dispersible in water but not soluble in any organic solvent and with an even better resistance to solvents than enamels based on terephthalic acid polyesters and a number of new synthetic resin-based rust protection and thixotropic enamels.

Polytetrafluoroethylene is the raw material used by the French firm of Gachot for the manufacture of machine parts. With the trade name of Gaflon, the French company's plastic has a temperature ceiling of 260°C. It is acid-proof, self-lubricating, has good electrical insulation powers and a lower temperature limit of −80°C, and is used either for machine parts, in conjunction with stainless steel, or for the manufacture of pipes, bands, plates, etc.

Among the exhibitors of chemical machinery and plant was one of the few British exhibitors in the chemical sector, R. H. Windsor, of Chessington, Surrey, with a display of plastics-processing machinery.

The more directly chemical plant consisted mainly of medium-size mixing and kneading machines, outstanding among which was a newly-developed mixer of the Gustav Spangenberg Maschinenfabrik concern of Mannheim. Called the Desagglomerator, this machine—now patented both in Germany and abroad, mixes, disperses, dissolves, emulsifies, homogenises, pigmentates and humidifies, if necessary with vacuum operation. A similarly versatile machine was offered by the Henschel-Werke GmbH company of Cassel. The full range of this firm's 'Pentax' stirrers for the chemical industry covers vessel capacities of 20 litres for laboratory work to vessels containing 15,000 litres. A selection of smaller machines for laboratory purposes was on show at the stand of the Dutch firm N. V. Nautamix; these included the extra-small mixer Nauta-Lilliput with a working capacity of only ½ gallon.

Among displays not connected with plastics or synthetics interest lay in the showing by E. Merck of their brand-new oil-soluble light protection preparation Eusolex 3490 for gas chromatography and other branches of chromatography as well as a large range of vitamins and pharmaceutical raw materials, in the emulsion softening agents for the rubber industry based on the esters of fatty acids and displayed by the Schill and Seilacher concern and in the range of guanide salts ($CH_4O_3N_4$ and others) for the pharmaceutical industry shown by Süddeutsche Kalkstickstoff-Werke.

Without Market Research the U.S. Might Have Polythene 'Coming Out of its Ears'

CHEMICAL market research was recently likened to gambling on a horse race—only in the case of the former the gamble was not for $2, it would probably run between $2 million and $20 million. The gamble was not two or four minutes in duration, but would run from two to 20 years.

This was stated by Mr. Roger Williams, jr., president of the Roger Williams Technical and Economic Services Inc., Princeton, N.J., in a paper on market research and long-range planning that he presented at a meeting of the American Institute of Chemical Engineers recently.

In the past 10 years or so chemical market research by company personnel and by consulting firms had grown to the point where expenditures were now at about $5 million a year. In his definition of chemical market research, Mr. Williams did not include sales analysis or marketing research—both were short-range management tools.

Chemical market research was a long-range tool. It might result in an immediate decision to build a plant that could not be running for two years from the time of the market research report. Management was most interested in the probable markets for the first 10 years of operation of the plant in estimating its future profitability.

Chemical market research reduced the gamble on a new plant and it showed research management that a given objective might have a greater potential than another objective. It could also carry out another long-range planning function that many people overlooked.

The U.S. was becoming the major source of capital for investments in foreign countries throughout the free world. More and more the European Common Market and the Central American Common Market would have to be considered by U.S. chemical managements. They should study those markets, their economics, their development and decide whether or not to participate in them.

For instance, U.S. exports of polythene in 1958 exceeded the capacity of three major polythene plants. If those exports were cut off by installation of plants in the importing countries, the U.S. would "have polythene coming out of its ears." Hence the need to do market research on a global, or at least free world, basis.

Firms interested in foreign market research should not rely on their sales staffs for they were born optimists. They should send chemical market research men with experience in foreign countries. That would save them money, and give them a better study and analysis.

PEOPLE in the news

● MR. RICHARD FORT, M.P. for the Clitheroe division of Lancashire, who was killed in a motor accident on 16 May, took an honours degree in chemistry at Oxford and then studied industrial chemistry for two years in Vienna. In 1932 he joined Imperial Chemistries Industries as a junior works manager in their Cheshire alkali works and from 1938 to 1940 he was president of I.C.I. (New York) Ltd. He was one of the three lay members of the Medical Research Council and was chairman of the Parliamentary and Scientific Committee and a governor of the Imperial College of Science and Technology.

● MR. RAYMOND A. SILK, deputy director of the Packaging Centre, a member of Council of the Institute of Packaging and a director of Packaging Intelligence Ltd., left London on 21 May for a six week's fact-finding tour of the U.S. Nominated by the Institute of Packaging, he is the only British member of a team of ten experts briefed by the European Productivity Agency to study U.S. packaging developments and methods of consultancy in this subject.

● DR. P. H. SYKES, who has been appointed research and development director of the Reed Paper Group, is a New Zealander who after graduating at Victoria University worked in the N.Z. Government agricultural research laboratory and later gained his Ph.D. at King's College, London. He joined Imperial Chemicals Industries Ltd., in 1935 and in 1946 went to British Oxygen as chemicals development manager, ultimately becoming technical and research director. He was successively director of several subsidiaries and joined the main board in 1953.

Dr. P. H. Sykes

● MR. STANLEY J. DAVIES, chairman and joint managing director of A. Gallenkamp and Co. Ltd., was re-elected president at the recent annual meeting of the British Lampblown Scientific Glassware Manufacturers' Association. MR. P. J. EDWARD, managing director of Franco-British Co. Ltd., and MR. H. V. STOUT, managing director of H. Stout and Co. Ltd., were re-elected vice-presidents.

● MR. MAT SIMPSON and MR. DAVID BROOKE-BOULTON have been appointed area sales representatives of the International Synthetic Rubber Co. Ltd. Mr. Simpson, who is 27 and has been appointed to the Northern area was for a number of years chief chemist of Aerialite Ltd. Mr. Brooke-Boulton, aged 29, joined the company in March this year after six years in the laboratory and sales department of Revertex Ltd. He has been appointed Southern area representative.

● MR. R. P. H. GASSER, B.A., has been elected to an official fellowship and tutorship in chemistry of Corpus Christi College, Oxford, effective 1 October.

● MR. JOHN L. SWEETEN, M.A., B.Sc., M.I.Chem.E., A.R.I.C., has been appointed general manager of the chemical engineering division of A.P.V. Co. Ltd. He had been a project manager with Kellog International Corporation since 1954. Mr. Sweeten took his M.A. at Cambridge and B.Sc. at London and in 1949 was joint Moulton Medallist of the Inst. Chem. Eng. He is secretary of the Chemical Engineering Group of the S.C.I.

Mr. J. L. Sweeten

● Quickfit and Quartz Ltd. have appointed MR. A. G. MATTHEWS as work study engineer. Mr. Matthews will undertake consolidation of a work study department to investigate all work carried out by direct and indirect labour, plant layouts, organisation and methods, with a view to increasing existing efficiency.

● ING. PIERO GIUSTINIANI, administrator-delegate of Societa' Montecatini, Milan, has left Italy for the U.S. to study the possibility of further expansion of his company's activities.

● DR. M. A. PHILLIPS, F.R.I.C., principal of Dr. M. A. Phillips and Associates, consulting chemists and chemical engineers, has been elected to the Romford, Essex, Borough Council.

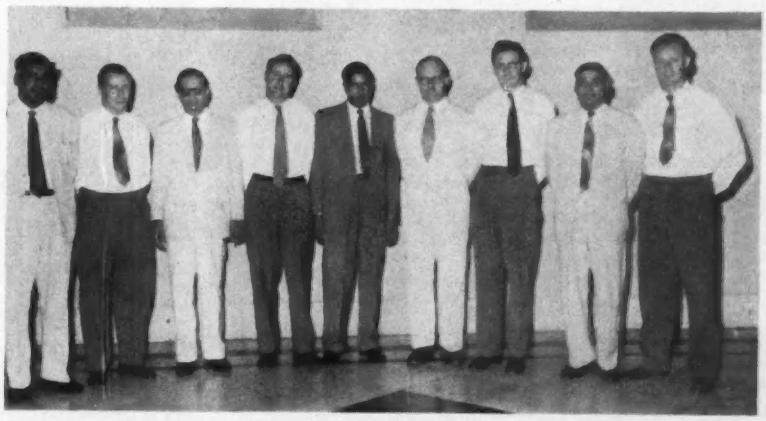
● MR. G. E. ROBERTSON has been appointed manager, sales and contracts, of the Meter, Relay and Instrument Division of the English Electric Company, Stafford, and MR. P. G. BEVIS has been appointed manager, sales and contracts, relay department.

Fundamentals of Polymerisation Processes

One of three new 10-day residential courses announced by the National College of Rubber Technology, London N.7, is on the fundamentals of polymerisation processes. The course, which will run from 6 to 15 July, is for the non-specialist. A course on basic rubber technology for sales, buying, costing and planning staffs will be held from 6 to 15 July, and another, on basic plastics processing, will be from 15 to 24 July.

Course on Structure of Organic Compounds

In a short course on the application of physical methods to the determination of structure of organic compounds at Norwood Technical College, Knight's Hill, London S.E.27, a series of three lectures on Fridays, 12, 19 and 26 June at 6 p.m., will deal mainly with infra-red and ultra-violet spectroscopy. On Saturday mornings, 13, 20 and 27 June, there will be visits to the laboratories of Smith, Kline and French Ltd. and the Wellcome Research Laboratories.



Products of Evans Medical Supplies Ltd., Liverpool, are being made under licence by Biological Products Private Ltd., Hyderabad, and being distributed in India by Evans Medical (India) Private Ltd. This photograph was taken during a recent visit to India of Mr. Maurice J. Smith, overseas trade director. It includes Mr. Smith, Dr. A. Worlock and Mr. G. T. Randal Davies of Evans Medical Supplies Ltd., Dr. D. V. K. Raju, Dr. Desai, Mr. P. Tirumala Rao and Mr. G. N. Raju of Biological Products Private Ltd., and Mr. J. S. Robison of Evans Medical (India) Private Ltd.

Commercial News

• I.C.I. Expand Trade With East Europe
• B.D.H. Overseas Sales Beat Home Trade
• British Xylonite Increase Dividend
• F. W. Berk Report Improved Sales

Imperial Chemicals Industries

In his chairman's speech to stockholders of Imperial Chemical Industries Ltd., Sir Alexander Fleck expressed the hope that the 1959 tax reductions were just one step towards bringing taxation down to somewhere near the level of some of British industry's competitors in Central Europe.

Some export markets remained difficult but others had been more open than they were in 1958. An interesting development had been a substantial expansion in what had been and still was a modest amount of trade with Eastern Europe. These countries could become valuable additional markets for a growing range of products.

Turnover in home and export markets was somewhat above the previous year's, but it was too early to forecast with confidence the likely outcome for the year.

Sir Alexander said two-thirds of the stock handed over under the profit-sharing scheme had been retained by the employees.

British Drug Houses

For the first time overseas sales of British Drug Houses Ltd. and subsidiaries exceeded home sales by a small margin in 1958. Although home sales of pharmaceutical products showed little improvement and margins narrowed, the laboratory chemicals division did well both at home and abroad.

Total sales rose by more than 11% to nearly £7 million.

As previously reported, group net profits rose from £199,960 to £288,991 and the dividend is 12% (11¼% equivalent).

The chairman, Mr. G. C. R. Ely, said it remained the board's policy to diversify still further as opportunity offered.

Negotiations were taking place for the acquisition of another concern like I. Rowland James, pharmaceutical wholesalers, which was acquired recently.

British Xylonite

Bigger group sales and a bigger share of profit attributable to the Xylonite company have enabled the British Xylonite Co. Ltd. to increase the dividend from 13% to 15% and yet plough back £161,817.

Group sales to external customers amounted to nearly £12 million against £11 million the previous year. Consolidated group profit at £1,023,877 was 6% lower than the record figure of £1,090,557 the previous year, but taxation was lower and the consolidated net profit of the group at £480,877 was slightly better. The Xylonite Company's share of the net profit was £307,520 (£277,408).

In his statement the chairman, Sir Laurence Merriam, said the Expanded Rubber Co.'s Onazote was used to insulate the terminal pipelines for the shipment of liquid methane from the U.S.

There was a further improvement in the operating efficiency of B.X. Plastics and a special feature was the smooth running of the polystyrene plant. They were continuing to spend large sums on research and development, especially in B.X. Plastics, where the expenditure was 2½% of sales.

Evans Medical

Evans Medical Supplies Ltd., who are to shorten their name to Evans Medical Ltd., are studying in detail two new fields of pharmaceutical manufacture.

In 1958, when they celebrated their 150th anniversary, sales and available surplus were records, though costs increased and margins were reduced.

As already announced (CHEMICAL AGE, 18 April) group net profit was £188,768 (£158,878) and the dividend is 12½% (same) plus a special distribution of 2d per 5s unit from capital reserves. A one-for-three scrip issue is proposed.

F. W. Berk

Group profit of F. W. Berk and Co. Ltd. for 1958 was £190,344, compared with £149,371 the previous year. Taxation was £203,339 (£209,513). A final dividend of 4½d makes a total for the year of 6d a share.

The chairman, Mr. A. D. Berk, said sales had been almost maintained last year. The improved profit was due in the main to savings from the concentration of manufacturing activities. After a sluggish start to the current year sales had picked up and he expected profits to be maintained.

Abbey Chemicals Ltd. had earned a modest net profit in 1958 and he expected it to do well in 1959. Detarex Ltd. had done well. Berk Exothermics Ltd. was still in the development stage and not yet a profit earner but should make more rapid progress backed by the new powder plant, which came into production at the end of February.

Boots Pure Drug Co.

With a final of 10%, Boots Pure Drug Co. Ltd. are bringing the dividend for the year ended 31 March to 16%. This dividend on the ordinary shares as increased by a one-for-four scrip issue is equivalent to 20% on the old capital, compared with 18% last year, and is the sixth successive increase in the effective payment.

After allowing £350,000 for stock losses arising from purchase tax changes in the Budget, group profit amounts to £4,997,839 before tax, against £5,057,350. Tax takes £2,555,598 (£2,962,174) leaving a net profit of £2,442,241 (£2,095,176).

Market Reports

DEMAND FOR VARIED RANGE OF CHEMICALS

LONDON There has been a steady flow of new home business for a wide range of chemicals, although the movement, against contracts, to the main industrial consumers has experienced the usual holiday interruption. Agricultural chemicals continue in good demand.

Prices, on the whole, are firm and unchanged with export quotation keen in the face of competition.

Most of the coal tar products are in steady request with prices firm at recent levels.

MANCHESTER As usual at this time of the year trade conditions on the Manchester market for heavy chemical products during the past week have been extremely quiet. Deliveries generally to the consuming end against existing commitments have been on a restricted scale, and inquiries as well as actual new business has also been affected by the holiday conditions. An early return to normal trading in most sections of the market is looked for, though in the case of fertiliser materials seasonal quietness over the next few months may be expected.

GLASGOW Prices during the past week remained more or less firm, and over all, the level of business on the Scottish Market continued to show a fair volume of activity, except from those sections of industry where improvement would be welcomed.

Once again, the bulk of demands were mostly against current requirements, but there was a fair percentage of forward bookings placed. Quantities were maintained, and the range of chemicals quite varied.

The position in regard to agricultural chemicals continues to improve, and the past week has shown quite a volume of activity, both in demands and inquiries.

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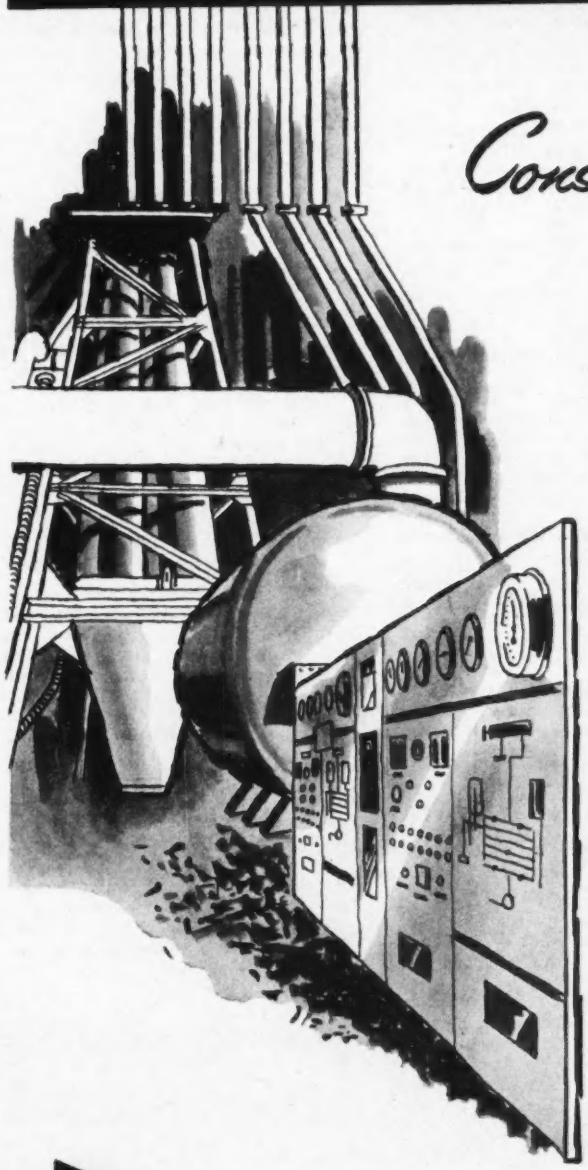

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TRADE NOTES

Colour-Mix Compounds

A new concept of colouring p.v.c. extrusions introduced by Bakelite Ltd., will be shown on Stand No. 35 at the International Plastics Exhibition on 17-27 June.

The new system is based on the use of self-coloured Vybak colour-mix compounds. These compounds are supplied (in the form of dice) to users of p.v.c. compounds in a range of basic colours, so that the user can produce a wide variety of shades by varying the proportions of each colour in the mix.

By stocking the base colours in Vybak colour-mix compounds, extrusions can be made immediately in many colours.

£3.5 Million Mexican Order

Mr. G. V. Sims, managing director of British Oil Equipment Credits Ltd., the newly formed subsidiary of the Council of British Manufacturers of Petroleum Equipment, has successfully negotiated an agreement with Petroleos Mexicanos (Pemex) for the supply of £3.5 million worth of petroleum equipment in connection with the new extension to the Minatitlan Refinery in Mexico. Pemex are shortly opening an office in London, under the direction of Senor I. A. Torres.

New Tri-Butyl Phosphate Plant

Albright and Wilson's new tri-butyl phosphate plant at Oldbury, near Birmingham, has been completed and will go into production shortly.

Agents for Polypenco Plastics

Polypenco Ltd., Welwyn Garden City, manufacturers of engineering industrial plastics, have appointed the following new agents: Northwest England—The Norman Wrigley Co. Ltd., 366 Stretford Road, Manchester 15; Scotland—Durward Clarkson & Co. Ltd., 137a St. Vincent Street, Glasgow C.2.

Uses of Titanium

The latest leaflet in the I.C.I. Metals Division's series dealing with the use of titanium in chemical plant is 'Titanium for Compressor Valve Plates and Springs'.

Points made are that because titanium combines a high strength-to-weight ratio with superlative corrosion resistance it is suitable for valve plates and springs in a much wider range of operating conditions than has been practicable hitherto and that long service life allows high initial cost to be recovered quickly.

Mechanical Seals for Refinery

Flexibox mechanical seals have been fitted to 156 rotary shaft pumps and mixers at Esso's Fawley refinery. So far more than 176 of these seals—two seals are required on some units—have been supplied for use on equipment manufactured by W. H. Allen Sons and Co., Campbell and Co., Hayward-Tyler and Co. Ltd., Pulsometer Engineering Co. Ltd. and Sigmund Pumps Ltd.

The pumps and mixers are handling products including cuprous ammonium acetate, acetic acid, caustic soda, butane, ethylene, naphtha, MEA, propane, fuel oils, gasoline and sea water. Operating temperatures extend from −140°F to 500°F and stuffing box pressures cover an equally wide range.

Mond Nickel Co. Exhibition

A private exhibition is being staged by the Mond Nickel Company at the Cutlers' Hall, Sheffield, from 2 to 5 June, featuring the properties of nickel, nickel-containing materials, the platinum metals and S.G. iron.

Control Instruments

Leaflet 103 issued by James Gordon and Co. Ltd., combustion engineers and boiler control specialists, Dalston Gardens, Stanmore, Middlesex, gives a complete list of their equipment, the range of which has considerably increased since the power plant division of Elliott Brothers (London) Ltd. became part of their organisation.

Leaflet 180 deals with the Elliott CO_2 meter, which is offered as an alternative to the Mono recorder, the Elliott CO_2 instrument being of the thermal conductivity type and the Mono CO_2 recorder operating on the chemical absorption principle.

O.C.C.A. Display for Scotland

British Titan Products Co. Ltd. are showing the panels, charts and paint formulations they prepared for the 1959 O.C.C.A. exhibition at the Scottish Building Centre, 142 Sauchiehall Street, Glasgow, on 27 and 28 May.

Limitation of Arsenic in Food

On 10 August 1959 regulations relating to the arsenic content of food will come into operation in England and Wales. The regulations lay down statutory limits for the arsenic content of food and beverages imported, sold or intended for sale for human consumption. The regulations limit with certain exceptions the arsenic content of ready-to-drink beverages to 0.1 parts per million and of foods to 1.0 p.p.m.

DIARY DATES

WEDNESDAY 27 MAY

S.C.I.—London: 14 Belgrave Sq., S.W.1, 6.15 p.m. Food Group A.g.m. Chairman's address, 'Food and the ordinary man', by Dr. J. R. Nicholls.

THURSDAY 28 MAY

Institute of Metals—London: 17 Belgrave Sq., S.W.1. 6.30 p.m. 'The atomic mechanisms of fracture', by Prof. N. J. Petch.

S.A.C.—Luton: Coll. Further Educ., 6.30 p.m. 'The analysis of silicones and related organo-silicon compounds', by Mr. J. C. B. Smith.

S.C.I.—Road and Building Materials Group visit to the Building Research Station, Garston, near Watford.

FRIDAY 29 MAY

C.S.—Coventry: Courtaulds Ltd., 7 p.m. 'Polyethers', by Prof. G. Gee.

S.C.I.—Food Group summer tour to Sweden (to 8 June).

Government Support for Research in Water Supply

FINANCIAL aid to the Water Research Association is now being given by the Government, through its Department of Scientific and Industrial Research. This research association of the Water Industry has been in operation for just over three years and has been engaged on research into the detection of leaks in water mains, the coagulation process of water purification and the use of plastic pipes for water supply. During this time its income has increased from £11,000 to over £20,000 per year, but the research needs of the industry are now so pressing that it has been decided to double the annual subscription of the association and thereby 'earn' a £27,000 Government 'Grant in Aid'.

The Council of the Association has been strengthened by the appointment of Mr. J. E. Beddoe, assistant secretary, Water Division, Ministry of Housing and Local Government, of Dr. B. A. Southgate, C.B.E., B.A., Ph.D., D.Sc., F.R.I.C., director, Water Pollution Research Laboratory, and of Mr. G. A. McMillan, Department of Scientific and Industrial Research.

D.S.I.R. has appointed Professor F. H. Garner, O.B.E., Ph.D., F.R.I.C., Director of Chemical Engineering Department, Birmingham University, and Mr. C. S. Tanner, M.Sc., M.I.Mech.E., Imperial Chemical Industries Ltd., Billingham Division (ammonia), as 'visitors'.

With anticipated income of £72,000 the Water Research Association is establishing its laboratories on the Thames in Buckinghamshire. There, with a staff of about 40, more than double the present number, it will be able to augment its research programme by the addition of filtration studies, investigations on the disposal and reclamation of sludge, and methods of biological and chemical analysis, methods of control of algal growth and to pursue some of the hydrological interests of the industry.

These decisions were taken at an extraordinary general meeting on 30 April at which Dr. B. K. Blount spoke on behalf of D.S.I.R. and Sir George McNaughton on behalf of the Ministry of Housing and Local Government, bringing with him the best wishes of the Minister for the success of the association.

Ubbelohde's New Types of Solid Conductors

AMONG exhibits at a conversazione held by the Royal Society at Burlington House, London W.1, on 14 May was one by Prof. A. R. Ubbelohde, Department of Chemical Engineering, Imperial College, London, showing new types of solid conductors of electricity based on graphite and on its crystal compounds with potassium and bromine.

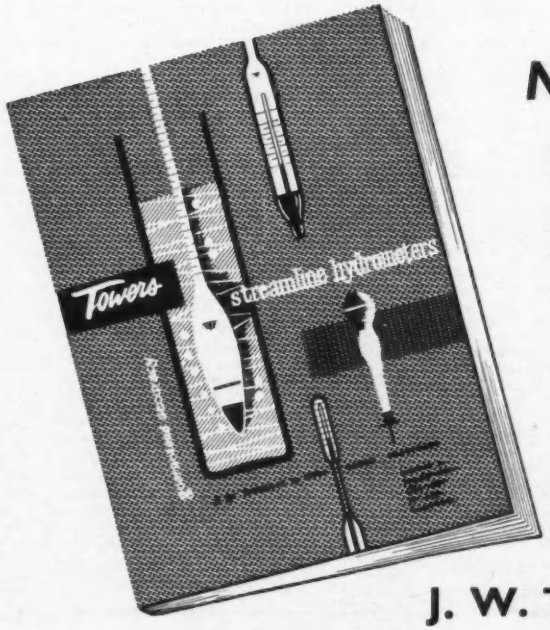
One section displayed the change of conductance of graphite in its two principal directions on forming crystal compounds with bromine and potassium. Static exhibits of crystal compounds and illustrations of natural and pyrolytic graphite were shown.

One of the crystal compounds, C_8K, has a specific resistance (a-axis) lower than that of metallic nickel. A live demonstration was given of the cyclic change in resistance of a sample on bromination and debromination.

The other section of the exhibit was concerned with the anistropic thermo-electric properties of graphite. A high-temperature graphite thermocouple was demonstrated incorporating a junction of polycrystalline and a-axis pyrolytic graphite. Values of thermo-electric power have so far been measured up to 2,100°C.

Agents for German Firm

Watson Brothers (London) Ltd., 12 Kingly Street, London W.1, have been appointed agents for the British Isles for Gehr. Raacke, Ottostrasse 57, Aachen, W. Germany, makers of electrolysis apparatus.



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NEW PATENTS

By permission of the Controller, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sales Branch), 25 Southampton Buildings, Chancery Lane, London W.C.2, price 3s 6d including postage; annual subscription £8 2s.

Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

ACCEPTANCES

Open to public inspection 1 July

Mild hydrofining of light virgin naphthas Esso Research & Engineering Co. **815 957**

α-Chloroglutaric anhydride. Du Pont De Nemours & Co. E. I. **815 960**

Separating into its constituents the reaction mixture derived from the cycloactatetraene synthesis. Badische Anilin- & Soda-Fabrik A.G. **815 961**

Acid addition salts of esters of 2-phenyl-2 (2-piperidyl) ethanol. Soc. Des Usines Chimiques Rhone Poulenc. **815 962**

Bacteriocidal hexamine compound. Farbenfabriken Bayer A.G. **815 770**

Gas filters. Wheelabrator Corp. **815 710**

Veterinary compositions containing organotin compounds.Metal & Thermit Corp. **815 818**

Piperazine-stannonium compounds. Metal & Thermit Corp **815 819**

Production of 0.0-dialkylphosphoric and 0.0-dialkylthionaphoric acid esters of substituted halogen-butenols. Badische Anilin- & Soda-Fabrik A.G. **815 965**

Dyestuffs of the auramine series which are soluble in alcohols. Badische Anilin- & Soda-Fabrik A.G. **815 966**

Dioxolane derivatives and process for their preparation. Soc. Des Usines Chimiques Rhone-Poulenc. **815 968**

Separation of mixtures of esters and alcohols. Badische Anilin- & Soda-Fabrik A.G. **815 774**

Therapeutic compositions containing 1-hexyl-3; 7-dimethylxanthine. Chemische Werke Albert. **815 969**

Open to public inspection 8 July

Recovery of sulphuric acid from waste acid. Minister of Supply. **816 290**

Production of a cementitious paste. Hatchard, M. Gallai-. **816 132**

Workable alloys of molybdenum and tungsten containing rhenium. Associated Electrical Industries. **816 135**

Sheets of thermoplastic material having a porous or cellular structure. British Celanese Ltd. **816 136**

Derivatives of formaldehyde condensation compounds of melamine and manufacture and use thereof. Ciba Ltd. **816 044**

Production of polymeric esters by ester-interchange reactions. Gevaert Photo-Producten N.V. **816 215**

Titanium-base alloys. Crucible Steel Co. of America. **815 974**

Manufacture of aliphatic dicarboxylic acids. Farbwerke Hoechst A.G. **815 983**

Treating alkylene polymers. Bataafsche Petroleum Maatschappij N.V., D.E. **816 292**

Apparatus for evaporating and circulating a liquid. Separator A.B. **816 216**

Hydrocinnolones and process for their manufacture. Ciba Ltd. [Addition to 788 393.] **816 108**

Refining crude silicon. Lonza Electric & Chemical Works Ltd. **816 047**

Production of polymers. Monsanto Chemical Co. **816 110**

Manufacture of steroids. Ciba Ltd. **815 987**

Resin treatment of textiles. Elöd, E. **816 330**

Liquid-gas separators. Muller, J. **816 331**

Manufacturing starch products. Scholten's Chemische Fabrieken N.V., W. A. **816 049**

Alkali hydroxides and in photographic developers containing them. Kodak Ltd. **816 220**

Preparing a gas suitable for the case hardening of steel. Regie Nationale Des Usines Renault. **816 051**

Manufacture of cellulose triacetate filamentary materials. British Celanese Ltd. **816 052**

Synthesis of caffeic esters of quinic acid and quinide. Farmaceutici Italia S.A. **816 053**

Sizing fibrous glass materials and the formation of composite articles therefrom. Union Carbide Corp. **816 056**

Elastomer compositions. Goodrich Co., B. F. **816 223**

Liquid-dispersing system. Ajem Laboratories Inc. **816 335**

Preparation of vitamin A esters. Eastman Kodak Co. **816 224**

β-Hydroxybutyric acid paraphenetidide and a process for its manufacture. Farbwerke Hoechst A.G. **816 225**

Solvent extraction of hydrocarbon oils. Esso Research & Engineering Co. **816 057**

Dibenzoate esters of aliphatic diols and process for their preparation. Union Carbide Corp. **815 991**

Preparation of hydrophobic siliceous material. General Electric Co. **816 337**

Saturated aldehydes. Distillers Co. Ltd. **816 151**

Polymeric bisthiuram disulphides and fungicides derived therefrom. United States Rubber Co. **816 341**

Mixed triglyceride composition. Hedley & Co. Ltd., T. **816 343**

Compositions including mixed triglycerides. Hedley & Co. Ltd., T. **816 344**

Vulcanisation of aqueous dispersions of rubber. Revertex Ltd. **816 230**

Separation of acetylene-free ethylene from gas mixtures. Lindes Eismaschinen A.G. Für G.m.b.H. **816 231**

Cement composition and method of preparation. Pennsylvania Salt Manufacturing Co. **816 233**

Separation of hydrohalic acids. Soc. D'electro-Chimie, D'electro-mettallurgie Et Des Acieries Electriques D'ugine. **816 154**

Epoxymethylol phosphorus derivatives. Albright & Wilson Ltd. **816 069**

Continuous catalytic polymerisation processes and processes for the production of catalysts therefor. Bataafsche Petroleum Maatschappij N.V., DE. **816 234**

Preparation of phenanthridine compounds. Boots Pure Drug Co. Ltd. [Addition to 767 588]. **816 236**

Preparing oligomycin and the oligomycin resulting from said process. Wisconsin Alumni Research Foundation. **816 070**

Corrosion resistant lubricant composition. Standard Oil Co. **816 237**

Electric conductors coated with modified alkyd resin compositions. Midland Silicones Ltd. **816 239**

Removal of fumes from gases. British Oxygen Co. Ltd. **816 001**

Veterinary compositions containing antibiotics. Imperial Chemical Industries Ltd., Beatson, E. P., and Floyd, J. C. **816 239**

Antibiotic, novobiocin, and preparation thereof. Pfizer & Co. Inc., C. **816 128**

Production of 1-(β-hydroxypropyl)- theobromine. Chemiewerk Homburg A.G. **816 299**

Bismuth salts and their preparation. Mining & Chemical Products Ltd. **816 300**

Furnace refractory linings. Imperial Chemical Industries Ltd. **816 003**

Process for producing opacifying effects on materials knitted from synthetic threads. Foerster, M., and Lehmann, A. [trading as Inhag Textilwerke Foerster M. Komm. Ges.]. **816 004**

Thiophosphoric acid esters and their production. Farbenfabriken Bayer A.G. **816 005**

Water emulsifiable compositions. Switzer Bros. Inc. **816 006**

Salicylates. Monsanto Chemicals Ltd. **816 245**

Fuel compositions. Bataafsche Petroleum Maatschappij N.V., DE. **816 306**

Production of ammonium persulphate and potassium persulphate. Food Machinery & Chemical Corp. **816 125**

Process of making diaryloxazole quaternary salts, diaryloxazole quaternary salts, made thereby, cyanine dyes containing a diaryloxazole nucleus made from such salts and photographic silver halide emulsions containing such dyes. Kodak Ltd. **816 014**

Preparation of cyclic dibasic acids. Henkel & Cie G.m.b.H. **816 248**

Preparation of acrylonitrile. American Cyanamid Co. **816 249**

Methods for the boron cementation of metallic articles by cathodic treatment in baths, consisting substantially of borates of monovalent and divalent metals, with boric anhydride. Soc. Metram Metallurgie Et Traitement Des Metaux. S.a.L. **816 081**

Emulsion polymerisation of unsaturated compounds. Hercules Powder Co. **816 252**

Production of benzaldehyde from toluene. Chemon Corp. **816 253**

Preparation of foamed bitumen. Shell Research Ltd. **816 254**

Method of catalyst manufacture. Sinclair Refining Co. **816 307**

Reduction of multivalent halides. National Distillers & Chemical Carp. **816 017**

Corrosion-resistant aluminium alloys. British Aluminium Co. Ltd. **816 182**

Production of transparent rubber articles from natural latex. Farbenfabliken Bayer A.G. **816 018**

Polymerisation of alkyd resin systems. United States Rubber Co. **816 019**

Methods of reducing unsaturated compounds. Philips Gloeilampenfabrieken N.V. **816 256**

Thiazolo-[5, 4-d]-pyrimidines and process for their manufacture. Ciba Ltd. **815 976**

Methods of reducing an unsaturated compound with an alkali metal or alkaline-earth metal. Philips Gloeilampenfabrieken N.V. **816 353**

Glass compositions and methods of making same. Owens-Illinois Glass Co. **816 161**

Production of coatings. Du Pont De Nemours & Co., E. I. **816 024**

Purifying aldehydes and ketones of the vitamin A series. Philips Gloeilampenfabrieken N.V. **816 264**

Manufacturing photographic material distinguishable in ultra-violet light. Soc. Per Azioni Ferrania. [Addition to 772 916] **816 026**

Soluble oil composition. Gulf Research & Development Co. **816 267**

Water-insoluble monoazo dyestuffs of the benzene-azo-benzene series. Sandoz Ltd. [Addition to 792 212.] **816 308**

Polymers of 3, 4-epoxycyclohexylmethyl 3, 4-epoxycyclohexanecarboxylates. Union Carbide Corp. **816 027**

Gasoline compositions. Socony Mobil Oil Co. Inc. **816 090**

17 α-Alkyl-17 β-hydroxyestran-3-ones and their preparation. Searle & Co., D. G. **816 091**

Halophosphate phosphor material. Westinghouse Electric International Co. **816 030**

Cycloalkylamides of *d*-lysergic acid and method for producing same. Semonsky, M., and Zikan, V. **816 273**

Process and product for improving the opacity of fibrous cellulosic products. Framalite. **816 031**

Bonding of non-metallic refractory members to metal members. General Electric Co. **816 032**

Drier for loose material, particularly granular material. Fleissner, H., Fleissner, H., and Fleissner, G., [trading as Fleissner & Sohn Maschinenfabrik]. **816 094**

Preparation of metal phosphate catalyst. Dow Chemical Co. **816 034**

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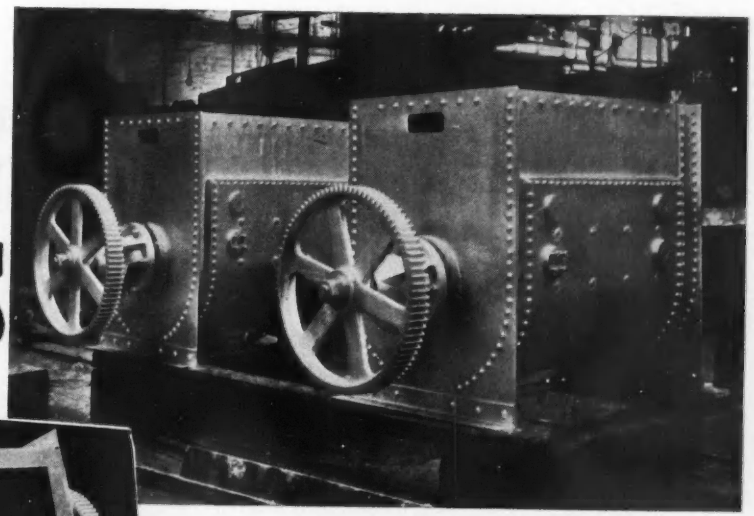

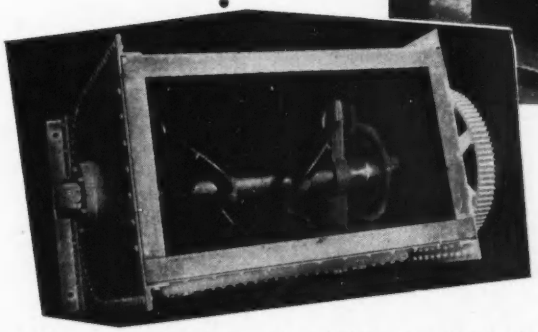

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